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Chemical Residues in Irish Farmed Finfish, 2012-2014

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Marine Institute
Foras na Mara

Mission Statement

The Marine Institute is the national agency which has the following functions:
'to undertake, to coordinate, to promote and to assist in marine research and development and to provide services related to marine research and development, that in the opinion of the Marine Institute will promote economic development and create employment and protect the environment'
Marine Institute Act 1991

Our Vision

A thriving maritime economy in harmony with the ecosystem and supported by the delivery of excellence in our services.

Acknowledgments

The following organisations and people within them, made valuable contributions in assisting with the delivery of 2012, 2013 and 2014 residue monitoring programme for farmed finfish:

Department of Agriculture, Food & the Marine
Food Safety Authority of Ireland
Sea Fisheries Protection Authority
The ongoing co-operation of the aquaculture industry

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CHEMICAL RESIDUE IN IRISH FARMED FINFISH 2012-2014

Report on monitoring of aquaculture undertaken in accordance with Council Directive 96/23/EC of 29 April 1996 *on measures to monitor certain substances and residues thereof in live animals and animal products.*

JULY 2015

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Summary

The Marine Institute carries out monitoring of chemical residues in aquaculture in accordance with Council Directive 96/23/EC of 29 April 1996, *on measures to monitor certain substances and residues thereof in animals and animal products,* also known as *the Residues Directive.* This is carried out on behalf of the Department of Agriculture, Food and the Marine (DAFM). For the aquaculture sector, the Sea Fisheries Protection Authority (SFPA) with technical support from the Institute is responsible for residue controls on farmed finfish on behalf of the national residue monitoring plan. Annually, the National Residues Control Plan (NRCP) for Aquaculture is prepared by the Institute and this sets out the monitoring requirements for residues in animal products in required by the directive. The main objectives of the National Residue Control Plan for Aquaculture is to ensure farmed fish are fit for human consumption; to provide a body of data showing that Irish farmed fish is of high quality; to promote good practices in aquaculture; and to comply with EU Directive 96/23/EC.

Based on production tonnage, the following species for the period of 2012 to 2014 were sampled and tested: Atlantic salmon (*Salmo salar*), freshwater and sea reared trout (*Oncorhynchus mykiss & Salmo trutta*). In excess of 2,141 tests and a total of 4,972 residue measurements were carried out over this three year period. Tests were carried out for banned substances such as growth promoters and other unauthorised substances such as malachite green, which should not be present. Harvest fish were also tested for authorised veterinary treatments such as antibiotics and sea lice treatments, environmental contaminants such as trace metals, polychlorinated biphenyls and organochlorine pesticides, to check for compliance with Maximum Residue Levels (MRL) where available.

As in previous years, **no non-compliant results** were reported in the surveillance monitoring programme for farmed finfish during the period 2012 to 2014. Overall, in recent years the outcome for aquaculture remains one of consistently low occurrence of residues in farmed finfish, with 0.23% non-compliant results from routine targeted monitoring in 2004, 0.09% in 2005 and **one of full compliance with 0% non-compliant target residue results for the period 2006-2014.**

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1.0 BACKGROUND

All European Member States have a responsibility to monitor the use of veterinary medicines in food producing animals, to ensure that produce from these animals do not contain residues that could be harmful to consumers. Surveillance monitoring is implemented in accordance with Council Directive 96/23/EC of 29 April 1996, *on measures to monitor certain substances and residues thereof in animals and animal products*, otherwise known as the Residues Directive, which requires Member States to put in place national plans (National Residues Control Plan - NRCP) for the monitoring of certain chemical substances and residues in a range of food producing species and products e.g. cattle, pigs, sheep, farmed finfish.

The Marine Institute monitors aquaculture finfish for the presence of chemical residues in accordance with Council Directive 96/23/EC on behalf of Department of Agriculture Food and Marine (DAFM). For the aquaculture sector, the Sea Fisheries Protection Authority (SFPA) with technical support from the Marine Institute is responsible for residue controls on farmed finfish on behalf of the national residue monitoring plan. The NRCP for Aquaculture in Ireland is specifically for farmed finfish and forms part of the overall National Residue Control Plan. The main objectives of the NRCP for Aquaculture is to ensure farmed fish are fit for human consumption; to provide a body of data showing that Irish farmed fish is of high quality; to promote good practices in aquaculture and to comply with EU Directive 96/23/EC.

2.0 IMPLEMENTING THE DIRECTIVE

DAFM, SFPA, Food Safety Authority of Ireland (FSAI) and the Marine Institute implement the Directive as it pertains to aquaculture; outlined below is an overview of their roles for aquaculture and Table 1 gives a summary of each department and agencies' role with respect to the NRCP for aquaculture:

Figure 1: Agencies & Department involved in Residue Monitoring Programme for Aquaculture

- **Department of Agriculture Food and the Marine (DAFM)**

DAFM implements the overall residues controls in Ireland with the DAFM Veterinary Inspectors (VIs) who carry out routine inspections on finfish farms. These inspections are carried out to verify compliance with Fish Health, Animal Remedies, Feedstuffs, Animal-By Products and Food hygiene regulations. If an issue or non-compliance arises from a routine inspection, DAFM VIs will conduct a follow-up investigation. Assistance may be required from the Marine Institute with respect to sampling and analysis and from the SFPA in closing out the follow-up.

- **Food Safety Authority of Ireland**

The Food Safety Authority of Ireland co-ordinates the activities of the various departments and agencies involved in delivering this programme.

Table 1: Department and Agencies Roles

Department of Agriculture Food and the Marine
Implements the overall residues controls in Ireland
Food Safety Authority of Ireland
Coordinates the activities of the departments and agencies involved
Sea Fisheries Protection Authority
Responsible for ensuring compliance with the Directive for finfish aquaculture
Marine Institute
Implements the surveillance monitoring programme for farmed fish. Is the official laboratory for residue sampling and analysis and is the national reference laboratory (NRC) for a number of substances in Aquaculture
DAFM Veterinary Inspectors
Carry out routine on-farm inspections to verify compliance with various regulations including fish health, animal remedies, feedstuffs etc.

- **Sea Fisheries Protection Authority (SFPA)**

SFPA is responsible for ensuring compliance with the Residues Directive for Finfish Aquaculture and for the follow-up of samples that are non-compliant under the NRCP. If a non-compliant result were to arise from sampling under the NRCP the SFPA will carry out a follow-up investigation with the assistance of the Marine Institute to carry out sampling and analysis. Assistance may be required from DAFM VIs in closing out the follow-up investigation.

- **Marine Institute**

The Marine Institute is the official laboratory for residue sampling and analysis of farmed finfish under the NRCP. The Institute aids and assists the SFPA in follow-up investigations and provides scientific advice as required. Marine Institute officers are authorised under Section 10(1) of the Animal Remedies Act, 1993. The Institute is also a National Reference Laboratory (NRL) for certain substances under the overall NRCP (see section 8.3).

3.0 THE NRCP FOR AQUACULTURE

Annually, the Marine Institute prepares the NRCP for Aquaculture[1], which is reviewed and finalised by SFPA, FSAI and DAFM. This sets out the species, sample numbers, target substances and analytical methods in line with the specific requirements of Directive 96/23/EC. The NRCP once agreed is then submitted to the European Commission (EC) for approval. Figure 2, details the Annual Cycle of National Residue Control Programme for Aquaculture.

The national legal basis for the Residue Monitoring Plan is provided for in the Animal Remedies Act, 1993 and other relevant legislation in particular, the Control of Animal Remedies and their Residues Regulations, 2009. For further details of the main legislation and monitoring of the Irish finfish aquaculture industry reference the legislation section of this document.

As with other farmed animals, farmed finfish can be subject to disease and infestation which can have animal welfare, environmental and commercial implications. The scope of this testing under the NRCP is comprehensive covering the following broad categories outlined in Table 2.

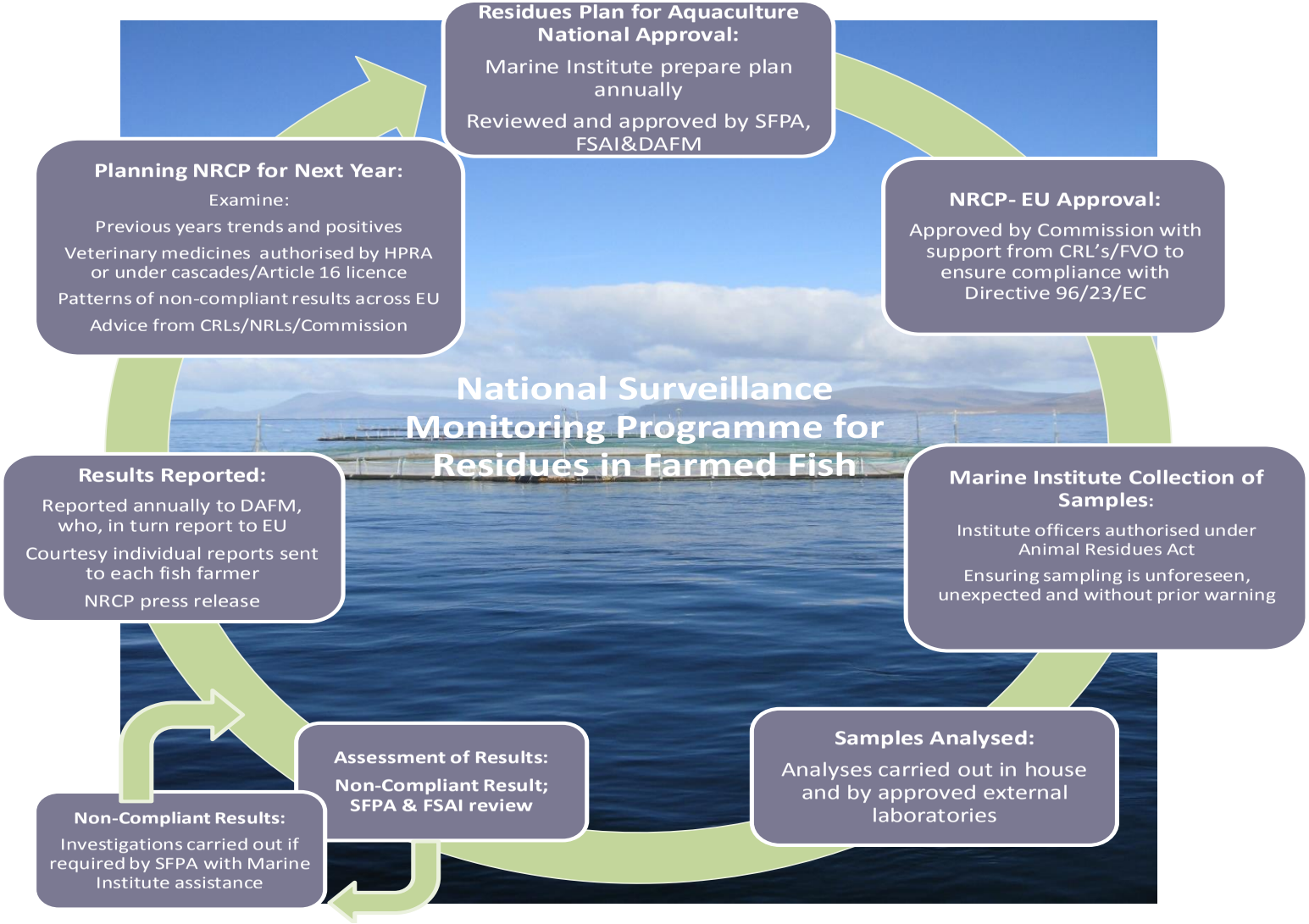
Table 2: NRCP testing categories

Category	Details
Banned	These compounds should not be present as no safe limit can be set for their residue e.g. steroids, chloramphenicol, nitroimidazoles
Authorised	Authorised medicines which may be used in aquaculture and should be below statutory limit (i.e. Maximum Residue Limit – MRL*) e.g. Sea lice treatments- emamectin, deltamethrin
Unauthorised	These compounds should not be present as these treatments should not be used in aquaculture. e.g. malachite green
Environmental contaminants	Certain contaminants can be introduced inadvertently which may accumulate in fish e.g. polychlorinated biphenyls (PCBs), organochlorine pesticides (OCPs), heavy metals

***MRL is the maximum concentration allowable in the edible portion of the animal which should not be exceeded at the time of harvest.**

[1] Reference Appendix 3 for 2012, 2013 & 2014 NRCP for Finfish

Figure 2: Annual cycle of National Residue Control Programme for Aquaculture



The substances are classed into two categories: Group A and Group B

Group A:

Group A substances are **banned substances** and should not be present in farmed finfish. These can be categorised as the following:

- A3 Steroids such as beta-oestradiol and methyltestosterone which occur naturally but also could be used for growth promotion
- A6 compounds such as nitrofurans and nitroimidazole which are antibacterial drugs; chloramphenicol which has a broad spectrum of antibiotic properties

A Maximum Residue Limits (MRL) cannot be established for these substances because of their toxicity; therefore, these substances are banned and should not be detected.

Group B:

Group B substances are classed into three categories: **unauthorised substances, authorised substances** and **environmental contaminants**. As with other intensively farmed animals farmed finfish can be subject to disease and infestation which can have animal welfare, environmental and commercial implications. Similar procedures are in place for the treatment of farmed finfish as for other farmed animals, with approved veterinary medicines administered under the direction of a veterinarian. These include antibiotics and anthelminthics to prevent or treat disease or infestation e.g. antibacterial agents, antifungal agents, antiparasitic treatments. To ensure compliance with Maximum Residue Limits (MRLs), operators are obliged to adhere to the specified withdrawal periods after treatment before harvesting.

In terms of environmental contaminates, fish including farmed finfish, can accumulate trace metals and persistent organic pollutants (POPs) from the environment or the feed. Seafood can be an important contributor to the dietary intake of POPs and mercury; therefore these contaminants are also examined. For further details on environmental contaminants see section 6.0.

Details are given in Table 3 of what group substances are tested for in the NRCP for aquaculture and a breakdown of the compounds that were tested for in the 2012 to 2014 plan outlined in Table 4.

Table 3: List of substances included in the NRCP for farmed finfish.

Group A–Substances having anabolic effect , banned		Group B- Veterinary drugs, unauthorised substances and contaminants	
A3	Steroids	**B1**	Antimicrobials (Antibacterial)
A6	Compounds included in Annex IV of Council Regulation 2377/90/EC	**B2a**	Anthelmintics (Antiparasitic)
		B2c	Pyrethroids
		B2f	Other pharmacologically active substances
		B3a	Organochlorine compounds
		B3c	Chemical elements
		B3d	Mycotoxins
		B3e	Dyes

Table 4: List of substances included in the 2012-2014 Residue plan for farmed finfish

Group A–Substances having anabolic effect, banned		Group B- Veterinary drugs, unauthorised substances and contaminants			
A3	Steroids: Methyltestosterone 17 ß-Oestradiol	**B1**	Antimicrobials (Antibacterial): Quinolones Tetracyclines Sulphonamides Florfenicol	**B3a**	Organochlorine compounds: PCBs OCPs
A6	Compounds included in Annex IV of Council Regulation 2377/90/EC: Chloramphenicol Nitrofurans Nitroimidazoles	**B2a**	Anthelmintics (Antiparasitic): Ivermectin Emamectin B1a Doramectin*	**B3c**	Chemical elements: Lead Cadmium Mercury
		B2c	Pyrethroids: Cypermethrin Deltamethrin	**B3d**	Mycotoxins: Aflatoxin
		B2f	Other pharmacologically active substances: Teflubenzuron Diflubenzuron Corticosteroids	**B3e**	Dyes: Malachite Green Leuco Malachite Green Crystal Violet* Leuco Crystal Violet* Brilliant Green* Victoria Blue*

***2014 analysis only**

3.1 VETERINARY MEDICINES

The overall national responsibility for the regulation of the veterinary medicines marketed in Ireland is with DAFM. The Health Products Regulatory Authority (HPRA), formerly known as Irish Medicines Board, is responsible for authorisation and licencing of animal remedies product. In addition to the authorised product license, DAFM also implements an 'exceptional' licensing regime for the importation of animal remedies authorised in another Member State in 'Certain Health Situations'; Regulation 16 S.I. 786 of 2007 and under the 'Cascade Provision'; Regulation18 S.I. 786 of 2007. The 'Cascade', is an exceptional mechanism provided for in EU and National legislation. This is designed to deal with situations where there is no authorised product to treat a particular condition in an animal, the emphasis therefore is on avoiding unacceptable suffering in an animal.

Some veterinary substances are used to control sea lice infestations of farmed salmon

Figure 3: Sea lice on fish

A particular issue for the finfish aquaculture sector is the requirement to control ecto-parasitic sea lice (*Lepeophtheirus salmonis, Caligus elongatus)* infestations on fish farms (Fig. 3). Sea lice levels are monitored on fish farms with a requirement to treat if trigger levels are exceeded; further information on results for sea lice inspections carried out by the Marine Institute in 2014 is available in O'Donohoe *et al*, 2015. Targeted treatment regimes can include synchronous treatments, i.e. "Single Bay Management". Further information is available in: "A strategy for the improved pest control on Irish salmon farms" (DAFF, 2010), which outlines a comprehensive range of measures to provide for enhanced sea lice control and in "The Farmed Salmonid Health Handbook" (Marine Institute, 2011), which assists producers in establishing a framework which will protect animal health and promote fish welfare on Irish farms. Table 5 details the veterinary medicines that are available for use in Ireland in 2014 for farmed finfish. Note in 2007 the Marine Institute carried out a review on veterinary treatments and other substances in Finfish Aquaculture in Ireland for South Western River Basin District (Marine Institute, 2007).

Table 5: Veterinary medicines authorised for use in Finfish 2014^

Medicine	Active Ingredient	Treatment	Group	Licensing Status
AMX ®	Deltamethrin	Bath	Pyrethroid	Full Medical Authorisation
Calicide ®	Teflubenzuron	In-Feed	Insect Growth regulator	Full Medical Authorisation
Aqualet ®	Oxytetracycline Hydrochloride	In-Feed	Antibiotic	Full Medical Authorisation
Maracycline ®	Oxytetracycline Hydrochloride	In-Feed	Antibiotic	Full Medical Authorisation
Excis ®	Cypermethrin	Bath	Pyrethroid	Full Medical Authorisation
Slice ®	Emamectin Benzoate	In-Feed	Avermectin	Full Medical Authorisation
Pyceze ®	Bronopol	Bath used on eggs	Antimicrobial	Full Medical Authorisation
Slice ®	Emamectin Benzoate	In-Feed	Avermectin	Full Medical Authorisation
Paramove ®	Hydrogen Peroxide	Bath	Oxidizer	Full Medical Authorisation
Florocol ®	Florfenicol	In-Feed	Antibiotic	Regulation18 ^^

^Further details on veterinary medicines reference report prepared by the Marine Institute for SWRBD March 2007
^^Certain veterinary medicines are authorised by DAFM in accordance with the provisions of national legi slation (Regulations 16 & 18, S.I. No. 786 of 2007).

3.2 ENVIRONMENTAL CONTAMINANTS

Farmed finfish can accumulate trace metals and persistent organic pollutants from their feed or the environment; therefore levels of these contaminants are examined in farmed finfish.

- **Trace metals**

Trace metals exist naturally in the environment and many, including chromium, copper, iron and zinc are essential elements for living organisms. Some trace metals such as mercury, lead and cadmium are not required for metabolic activity and can be toxic at quite low concentrations. These can also be introduced into the aquatic environment from activities such as mining, industry and municipal waste. Once in the aquatic environment these metals can be bioaccumulated. As part of the NRCP mercury, cadmium and lead are examined. Maximum levels for mercury, cadmium and lead in fisheries products are set out in Commission Regulation (EC) No 1881/2006 as amended *setting maximum levels for certain contaminants in foodstuffs*. For salmon and trout the levels specified are 0.3 mg kg^{-1} for lead, 0.05 mg kg^{-1} for cadmium and 0.5 mg kg^{-1} for mercury. These are taken as the "action levels" for the following report.

- **Persistent organic pollutants**

Persistent organic pollutants (POPs) are primarily anthropogenic toxic substances that are persistent in the environment. They can be transported long distances in air and water and consequently tend to be globally ubiquitous pollutants. They bioaccumulate in animal tissue, especially fatty tissues such as oily fish and may biomagnify through the food chain. Seafood is one of the key dietary sources for humans. Examples of POPs include polychlorinated biphenyls (PCBs), organochlorine pesticides (OCPs) such as DDT, dioxins, certain brominated flame retardants and perfluorinated compounds. Consequently POPs are widely monitored in seafood to ensure consumer safety. Measures to control or phase out POPs have been implemented at national, regional and global scales over recent decades (OSPAR 2010, EPA 2012) and environmental concentrations of many POPs are generally decreasing, although given their persistence this is a slow process. POPs are found in the tissue of farmed fish, primarily due to uptake from fish feed, and this is associated with the POP burdens in the wild fish oil/meal component of the feed (Berntssen et al. 2010).

An assessment of contaminants and residues in Irish seafood (2004 -2008) was carried out by the Marine Institute (McGovern *et al*, 2011). This identified that Irish seafood (fish and shellfish) was compliant with standards. The report also determined that estimates of intake of mercury, hexachlorobenzene and dioxins and dioxin-like PCBs from seafood consumption for the average Irish adult seafood consumer accounted for less than one fifth of the individual (provisional) tolerable weekly intake ((p)TWIs) as established by the European Food Safety Authority (EFSA) for these substances. Moreover, the report highlighted the well-established health benefits of eating fish, especially oily fish such as salmon, herring and mackerel. More information on POPs in Irish seafood, including farmed finfish, is available in McGovern *et al*, 2011 and in FSAI (2013) report which was carried out in conjunction with the Marine Institute in 2010-2011 to survey a broad range of halogenated POPs in Irish fishery products. Six farmed salmon and two sea reared trout were included in this survey.

As part of the NRCP the following POPs are examined:

- **Polychlorinated Biphenyls (PCBs):**
  PCBs are a group of homologous man-made substances with a molecular structure comprising of a chlorinated biphenyl ring. PCBs are used in a wide range of industrial applications due to their thermal and electrical insulation properties. Despite being banned since the 1980s they are still being found in long life equipment such as electrical transformers and capacitors (EPA 2012). They are persistent environmental contaminants that accumulate in lipids and as such can be present at levels of concern in fish. PCBs can be divided into groups according to their toxicological properties e.g. dioxin-like PCBs, non dioxin-like PCBs. As part of the NRCP, it is primarily the following six non dioxin-like PCBs (NDL-PCB) which are monitored; PCB 28, 52, 101, 138, 153 and 180. These NDL-PCBs are routinely used as a monitoring indicator as they are generally presumed to be the most persistent in fish tissue and comprise about half of the amount of total PCB present in feed and food. Recent European legislation (Commission Regulation (EU) No 1259/2011 which came into force 1st Jan 2012 amending Regulation (EC) 1881/2006) has fixed maximum levels for dioxins, dioxin-like PCBs and non dioxin-like PCBs in foodstuffs. In the case of NDL-PCBs (also known as ICES PCB 6) the maximum level of 75 ug kg^{-1} wet weight has been set for the sum of these six congeners and samples analysed as part of the NRCP are consistently within these regulatory limits.

- **Organochlorine pesticides (OCPs):**
  OCPs are synthetic substances used for pest control that are persistent and widespread in the marine environment, despite their use being progressively phased out in recent decades. Chlorinated pesticides behave similarly to PCBs in the environment and do not have maximum concentrations in fish set by the EC. Due to their chemical properties (fat solubility) these substances bio-accumulate in fish tissue and also bio-magnify through the marine food chain with high levels especially found in marine mammals. A number of OSPAR contracting countries have set levels and Table 11 shows the available standards/guidance values in so far as Marine Institute is aware of these. All the samples analysed for chlorinated pesticides as part of the NRCP were below these levels.

4.0 METHODOLOGY

4.1 SAMPLING

Residue sampling is carried out by Marine Institute authorised sampling officers (Authorised under the Animal Remedies Act 1993) in accordance with Council Directive 96/23/EC. The Institute ensures that sampling is unforeseen, unexpected, without prior warning and that a strict chain of custody is maintained in accordance with Article 3 of Regulation 882/2004; *"Member States shall ensure that official controls are carried out regularly, on a risk basis and with appropriate frequency"* and Article 12 of Council Directive 96/23/EC; *"The checks provided for in this Directive must be carried out by the competent national authorities without prior notice"*.

Figure 4: The Residue Directive for Finfish aquaculture- Sampling process

The following species were sampled and analysed as part of the NRCP for 2012 to 2014 (Fig. 5**): Atlantic salmon (*Salmo salar*), freshwater and sea reared trout (*Oncorhynchus mykiss* & *Salmo trutta*)**. Typically, five individual fish were collected per farm by a Marine Institute authorised officer from a producer at either the processing plant or fish farm. Each fish was taken as an individual sample, however where an individual fish was not large enough to provide sufficient test material, a

number of fish were pooled to provide a sample. Samples were further subsampled as multiple tests were typically performed on individual samples.

Atlantic salmon (*Salmo salar*)

Rainbow trout (*Oncorhynchus mykiss*)

Brown Trout (*Salmo trutta*)

Figure 5: 3 species sampled as part of NRCP 2012-2014 (BIM, 2015)

Figure 6 shows the residues sampling lifecycle from a fish farmer's perspective. Figure 7-9 illustrates the locations of farmed finfish sites sampled in Ireland in 2012, 2013 and 2014.

Figure 6: Residues sampling life cycle

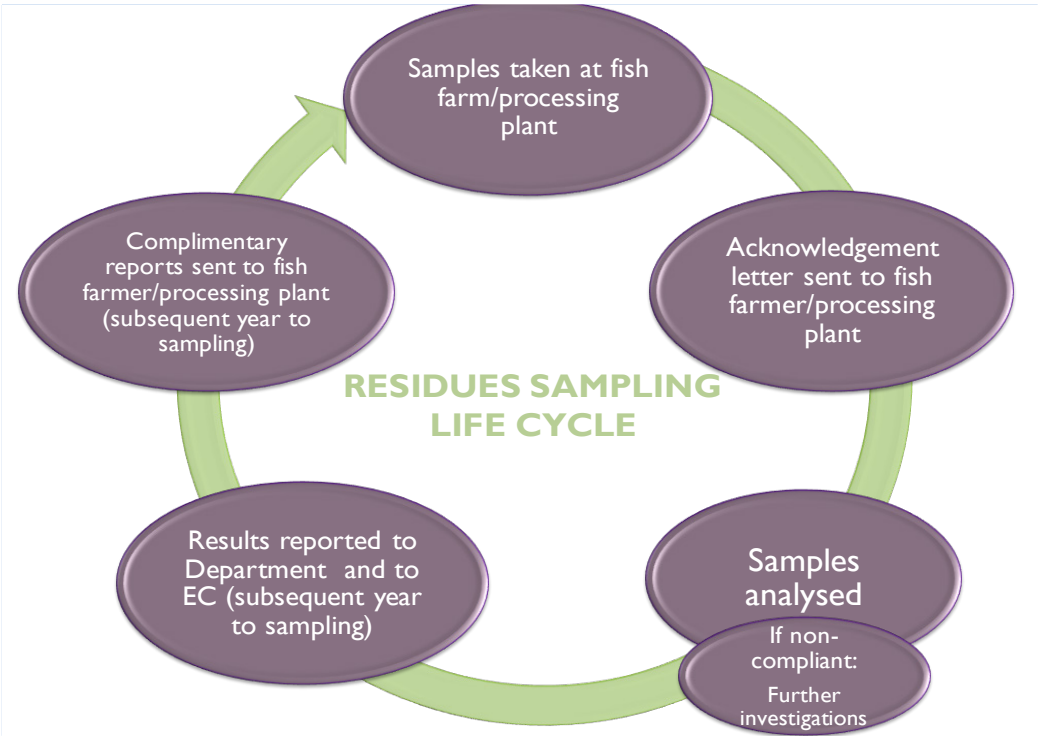


Figure 7: Locations of sites sampled as part of the residue programme in 2012. 1:3,157,319

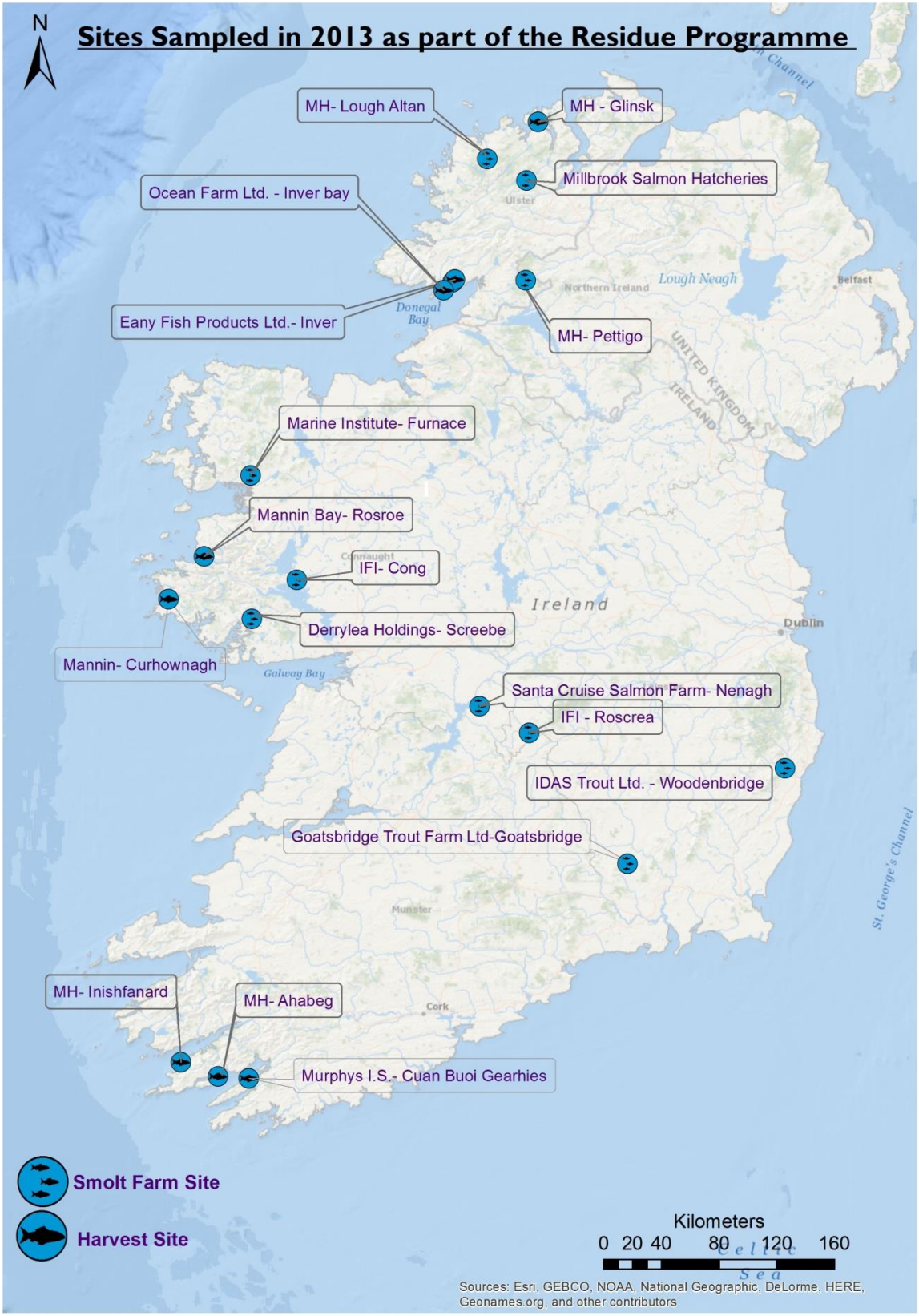


Figure 8: Locations of sites sampled as part of the residue programme in 2013. 1:3,157,319

Figure 9: Locations of sites sampled as part of the residue programme in 2014. 1:3,157,319

Samples were taken throughout the year in an effort to spread sampling across different sites and are taken in accordance with the National Residue Plan i.e.

- **Harvest:** Two thirds of the samples are taken at harvest stage which is aimed at ensuring compliance with the MRLs and for detection of illegal treatment (prohibited substances Group A and unauthorised substances Group B). These harvest samples are taken primarily at processing plants (Table 6) for salmon and sea reared trout, and 'on farm' for freshwater trout.

Table 6: 2012-2014 Active processing plants for salmon and sea reared trout

Eany Fish Products Ltd*
Irish Seafood Producers Group (ISPG)
Marine Harvest Ireland Ltd.
Murphy's Irish Seafood Ltd.
Ocean Farm Ltd.

***Closed June 2013**

- **Other Stages of Production (OSOP):** One third of the samples are taken 'on farm' at the smolt stage (Table 7) prior to going to sea for salmon and at OSOP for freshwater trout. This is aimed at detection of illegal treatment (prohibited substances/Group A and unauthorised substances /Group B3 (e)-Dyes).

Table 7: Autumn or spring smolts

Spring smolts	Autumn smolts
S1's : **Smolts introduced to the sea in March**	**S0's:** **Smolts introduced to the sea in November**
Details: • life cycle of the salmon when it changes from being a freshwater fish to a seawater fish. This process is known as smoltification. • fish are transported to the saltwater environment in the spring, which is approximately 15 months after hatching.	Details: • also known as $S_{1/2}$ • fish are exposed to manipulated photoperiods to hasten the onset of smoltification. Hence an S0 smolt is ready to go to sea during the Autumn/Winter, approximately 11 months after hatching.

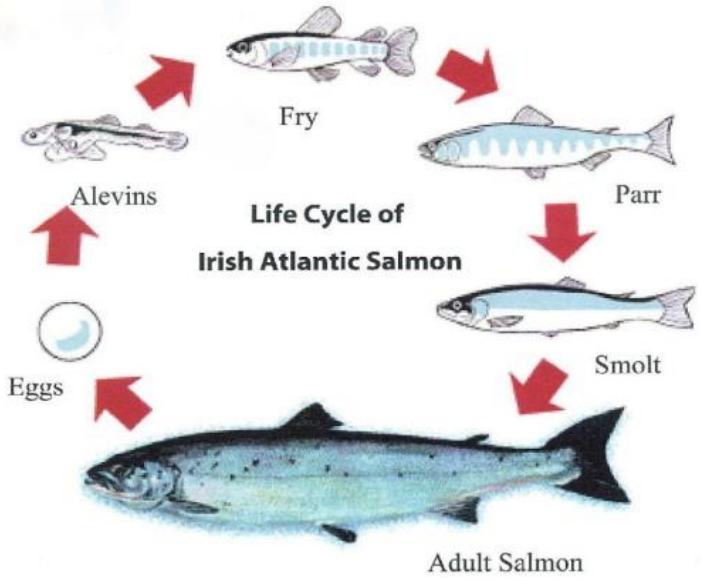


Figure 10: Life cycle of Irish Atlantic Salmon (Marine Institute, 2015)

Detailed below is a summary of the sampling activities for the period 2012 to 2014. See maps in figures 7-9 and appendix 4 for full list of sampling sites.

2012:
In 2012, a total of 169 target (surveillance) samples were collected from fish farms and processing plants in accordance with the NRCP for Aquaculture 2012 (Appendix 3) as follows:

- 57 target samples were taken at OSOP; 49 salmon smolts and 8 freshwater trout from twelve farms, for Group A and malachite green analysis.
- 112 target samples were taken at harvest which comprised of 89 farmed salmon, 15 sea reared trout and 8 freshwater trout. These harvest samples were collected during 23 sampling events (samples collected from a given site at a given time) throughout the year. Salmon were collected on eighteen occasions, freshwater trout twice and sea reared trout three times. Samples were collected from the same producers on a number of occasions due to the small number of active harvest sites in the given year.

2013:
In 2013, a total of 137 target (surveillance) samples were collected from fish farms and processing plants in accordance with the NRCP for Aquaculture 2013 (Appendix 3) as follows:

- 46 target samples were taken at OSOP; 38 salmon smolts and 8 freshwater trout from ten farms, for Group A and malachite green analysis.
- 91 target samples taken at harvest which comprised of 78 farmed salmon, 5 sea reared trout and 8 freshwater trout. These harvest samples were collected during 19 sampling events (samples collected from a given site at a

given time) throughout the year. Salmon were collected on sixteen occasions, freshwater trout twice and sea reared trout once. Samples were collected from the same producers on a number of occasions due to the small number of active harvest sites in the given year.

2014:
In 2014, a total of 136 target (surveillance) samples were collected from fish farms and processing plants in accordance with the NRCP for Aquaculture 2014 (Appendix 3) as follows:

- 45 target samples were taken at OSOP; 37 salmon smolts and 8 freshwater trout from ten farms, for Group A and malachite green analysis.
- 91 target samples taken at harvest which comprised of 78 farmed salmon, 5 sea reared trout and 8 freshwater trout. These harvest samples were collected during 19 sampling events (samples collected from a given site at a given time) throughout the year. Salmon were collected on sixteen occasions, freshwater trout twice and sea reared trout once. Samples were collected from the same producers on a number of occasions due to the small number of active harvest sites in the given year.

In 2014, during the first quarter of the year there was limited processing of harvest samples, therefore no harvest samples were taken in January and harvest sampling was confined to two fish farms for February, March and April.

Suspect Sampling:
No suspect sampling took place during the period 2012 to 2014 as there was no requirement for this arising from surveillance monitoring findings.

4.2 RESIDUE ANALYSIS, QUALITY SYSTEM & NRL

4.2.1 Residue Analysis

Prior to testing for a broad range of substances (Table 8), preparation of the samples is carried out upon arrival in the laboratory (Fig. 9),

On return to the Marine Institute, the integrity of the samples is checked to ensure chain of custody has been maintained. Samples are filleted, scored & homogenised. (Note: Archive sample is set aside and stored. This can be used where dispute over results occurs).The homogenised sample is sub- divided into several jars & stored in a locked freezer until analysis. The chain of custody remains intact from farm to final end of year report.

Figure 11: Sample preparation for NRCP samples

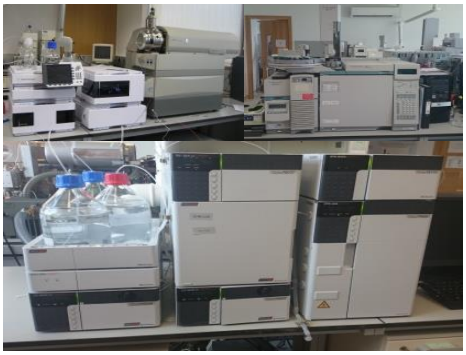
Table 8: List of analytical methods used for each substances in 2014 Residue Plan for farmed finfish

Group	Substance	Laboratory method	Group	Substance	Laboratory method
A3	Steroids: Methyltestosterone 17 ß-Oestradiol	 **ELISA** **ELISA**	**B2f**	Other pharmacologically active substances: Teflubenzuron Diflubenzuron Corticosteroids	 **LC** **ELISA**
A6	Compounds included in Annex IV of Council Regulation 2377/90/EC: Chloramphenicol Nitrofurans Nitroimidazoles	 **ELISA** **LCMSMS** **LCMSMS**	**B3a**	Organochlorine compounds: PCBs OCPs	 **GCHRMS**
B1	Antimicrobials (Antibacterial): Quinolones Tetracyclines Florfenicol Sulphonamides	 **2-plate** **Immunoassay**	**B3c**	Chemical elements: Lead Cadmium Mercury	 **GFAAS** **GFAAS** **CVAFS**
B2a	Anthelmintics (Antiparasitic): Ivermectin Emamectin B1a Doramectin*	 **LC**	**B3d**	Mycotoxins: Aflatoxin	 **LC**
B2c	Pyrethroids: Cypermethrin Deltamethrin	**GC**	**B3e**	Dyes: Malachite Green Leuco Malachite Green Crystal Violet Leuco Crystal Violet Brilliant Green Victoria Blue	 **LCMSMS**

This testing utilises a variety of modern analytical techniques (Fig. 12) and employs both screening and confirmatory techniques. Depending on the parameter, the testing is carried out in the Marine Institute laboratories or in approved external laboratories that meet the strict validation requirements (Appendix 1 for analytical methodology utilised).

A comprehensive quality assurance programme supports the monitoring programme as outlined below

Samples are weighed; homogenised with solvent to extract analyte(s) of test; followed by sample clean-up techniques such as liquid or SPE; analysed by various scientific analytical equipment.



Instrumentation used in analysis GC, HPLC, LCMSMS

Figure 12: Laboratory analysis for NRCP samples

4.2.2 Quality Assurance

Testing as part of the NRCP is carried out in-house in the Marine Institute and also by approved subcontracted laboratories. It is a requirement of the NRCP that all screening and confirmatory methods are validated to Commission Decision 2002/657/EC[2]. Commission Decision 2002/657/EC specifies the method performance criteria, the validation parameters required to demonstrate that an analytical method is fit for 'purpose' and the interpretation of results for analytical methods that are used for the Residue Directive. To underpin provision of high quality monitoring data, all analytical methods used by the Marine Institute for the NRCP are accredited to ISO17025. This is the international standard that ensures that analytical methods are fit for purpose. The Institute is subject to a range of audits including Irish National Accreditation Board (INAB) which is an annual audit against ISO17025. Table 9 lists the NRCP methods which the Marine Institute are accredited by the Irish National Accreditation Board (INAB) to ISO 17025 as detailed in Scope Registration Number 130T for 2014. Quality Control data for Marine Institute residue analysis in 2012 to 2014 is reported in Appendix 2.

[2] Commission Decision 2002/657/EC establishes criteria and procedures for the validation of analytical methods to ensure the quality and comparability of analytical results generated by official laboratories

Table 9: Accreditation to ISO17025

Scope Registration Number 130T	
• Ivermectin, Emamectin B1a , Doramectin	(CHE-8)
• Mercury	(CHE-32)
• Teflubenzuron , Diflubenzuron	(CHE-42)
• Cadmium	(CHE-85)
• Lead	(CHE-84)
• Cypermethrin, Deltamethrin	(CHE-25)
• Screening of Antibiotic Residues in Fish	(FHU-1)
• Screening of sulphadiazine	(FHU-119)
• Dyes: Malachite Green, Crystal Violet, Victoria Blue, Leuco Crystal Violet, Leuco Malachite Green, Brilliant Green	(CHE-167)
• Moisture %	(CHE-52)
Note:	
• When Collecting Samples the Laboratory Complies with Council Directive 96/23/EC	(CHE-6)

4.2.3 NRL

The Marine Institute is the National Reference Laboratory (NRL) for certain substances relevant to the NRCP (Table 10). In 2015, the Marine Institute took over the NRL duties from Veterinary Public Health Regulatory Laboratory (VPHRL) for chemical elements (mercury, lead and cadmium) in aquaculture products.

Table 10: Marine Institute National Reference Laboratory (NRL's)

Group B (2a)	Anthelmentics – Emamectin in Aquaculture only
Group B (2f)	Teflubenzuron & Diflubenzuron in Aquaculture only
Group B (3e)	Malachite Green and Leuco Malachite Green in Aquaculture only

4.3 RESULTS OF ANALYSIS

4.3.1 Interpretation of Results

Samples are deemed **compliant** (i.e. negative) if authorised compounds do not exceed the MRL prescribed by the EC in Commission Regulation (EU) No. 37/2010 Table 1 and if unauthorised substances are not detected above a defined analytical method decision limit. The MRLs and decision limits are specified in Table 11.

Samples are deemed **non-compliant** (i.e. positive) if concentrations of a given residue are confirmed to be present in excess of the MRL, where the MRL has been set. Where no MRL is set, (e.g. for banned substances including steroids and compounds listed in Table 2 of Commission Regulation (EU) No 37/2010 and for unauthorized substances), a decision limit (action level) is used and samples are reported as non-compliant where residues are confirmed to be present.

Generally, MRLs will not be exceeded if good husbandry practices are in place and the withdrawal periods are adhered to i.e. the animal is not slaughtered for a set period of time after treatment. A non-compliant result (i.e. a confirmed positive result) is assessed and reviewed by SFPA and FSAI and an investigation is carried out if required by SFPA with Marine Institute assistance.

Table 11: Maximum Residues Limits, Decision Limits and Guideline Values used for assessing compliance with Residues Directive

Parameter	Maximum Level or Decision Limit [6]	Source
Group A Compounds[1] : Methyltestosterone, 17β-Oestradiol, Chloramphenicol, Nitrofurans &Nitroimidazoles	These are banned substances and should not be detected.	
Ivermectin	0.4 μg kg^{-1}	Decision Limit[3]
Doramectin	0.4 μg kg^{-1}	Decision Limit[3]
Emamectin B1a	100 μg kg^{-1}	Maximum Residue Limit[2]
Cypermethrin	50 μg kg^{-1}	Maximum Residue Limit[2]
Deltamethrin	10 μg kg^{-1}	Maximum Residue Limit[2]
Teflubenzuron	500 μg kg^{-1}	Maximum Residue Limit[2]
Diflubenzuron	1000 μg kg^{-1}	Maximum Residue Limit[2]
Antibacterial Substances		
• **Sulphonamides**	100 μg kg^{-1}	Maximum Residue Limit[2]
• **Oxytetracycline (Tetracyclines)**	100 μg kg^{-1}	Maximum Residue Limit[2]
• **Oxolinic Acid (Quinolones)**	100 μg kg^{-1}	Maximum Residue Limit[2]
• **Flumequine (Quinolones)**	600 μg kg^{-1}	Maximum Residue Limit[2]
• **Sarafloxacin (Quinolones)**	30 μg kg^{-1}	Maximum Residue Limit[2]
• **Florfenicol**	1000 μg kg^{-1}	Maximum Residue Limit[2]
ICES PCB 6 [7]	75 μg kg^{-1}	EC Maximum Limit[8]
HCB	50 μg kg^{-1}	Norway (G)[4]
γ HCH	100 μg kg^{-1}	Finland (S)[4]
p,p'DDT and metabolites	500 μg kg^{-1}	Finland (S)[4]
Aldrin + Dieldrin	100 μg kg^{-1}	Finland (S)[4]
Endrin	50 mg kg^{-1}	Finland(S)[4]
Malachite Green	0.5 μg kg^{-1}	Decision Limit[3]
Leuco Malachite Green	0.5 μg kg^{-1}	Decision Limit[3]
Brilliant Green	0.5 μg kg^{-1}	Decision Limit[3]
Crystal Violet	0.5 μg kg^{-1}	Decision Limit[3]
Leuco Crystal Violet	0.5 μg kg^{-1}	Decision Limit[3]
Victoria Blue	0.5 μg kg^{-1}	Decision Limit[3]
Lead	0.3 mg kg^{-1}	EC Maximum Limit[5]
Cadmium	0.05 mg kg^{-1}	EC Maximum Limit[5]
Mercury	0.5 mg kg^{-1}	EC Maximum Limit[5]

1. Commission Regulation (EU) No 37/2010 (Table 2) and Directive 2008/97/EC: *Substances banned and should not be detected*
2. Commission Regulation No 37/2010 (Table 1) *on pharmacologically active substances and their classification regarding maximum residue limits in foodstuffs of animal origin*.
3. These compounds are not authorised for use in finfish, concentrations above the analytical methods decision limit are non-compliant.
4. OSPAR: *A compilation of standards and guidance values for contaminants in fish, crustaceans and molluscs for the assessment of possible hazards to human health*, Update 1993, JMP 17/3/10-E. (S) standard; (G) guidance value.
5. Commission Regulation (EC) No 1881/2006 *setting maximum levels for certain contaminant in foodstuffs and its amendments* Commission Regulation 629/2008/EEC and 565/2008/EEC.
6. Maximum Residue Limits and Decision Limits concentration are on a wet weight basis.
7. ICES PCB 6: sum of the following 6 CB congeners –PCB 28, 52, 101, 138, 153, 180.
8. Commission Regulation No 1259/2011 amending Regulation No. 1881/2006 as regards maximum levels for dioxins, dioxin-like PCBs and non dioxin-like PCBs in foodstuffs.

4.3.2 Summary of 2012-2014 results

As in the recent years, **no non-compliant results** for target samples were reported in the national monitoring programme for farmed finfish in 2012-2014. Outlined below is an overall summary:

- **2012:** in excess of 784 tests and a total of 1,596 residue measurements were carried out on 169 samples of farmed finfish for a range of residues: **all samples were compliant**

- **2013:** in excess of 651 tests and a total of 1,494 residue measurements were carried out on 137 samples of farmed finfish for a range of residues: **all samples were compliant**

- **2014:** in excess of 706 tests and a total of 1,882 residue measurements were carried out on 136 samples of farmed finfish for a range of residues: **all samples were compliant**

Summary target results from sampling of farmed finfish for residues from 2004 - 2015 are outlined in Table 12 and 13. Overall, the outcome for aquaculture remains one of consistently low occurrence of residues in farmed finfish, with 100% compliance for surveillance monitoring residue results for the period 2006-2015.

Table 12: Summary total target results for residue program for 2005-2009.

	2005	2006	2007	2008	2009
No. Target samples[1]	164 (105, 59)	162 (104, 58)	161 (103, 58)	162 (103, 59)	146 (98, 48)
Total Group A[2]	163/0	162/0	148/0	144/0	128/0
Total Group B[2]	164/0	162/0	161/0	162/0	146/0
Total No. of Results[3]	2251/2	2207/0	2219/0	2073/0	1750/0
% non -compliant results	**0.09**	**0**	**0**	**0**	**0**

[1]*Target samples (sampled at harvest, sampled at other stages of production)*

[2] *No. of samples tested/No. of samples non-compliant*

[3]*Total no. of results as samples taken for Group A and Group B substances are tested for multiple residue categories within each group*

Table 13: Summary total target results for residue program for 2009-2014

	2010	2011	2012	2013	2014
No. Target samples[1]	141 (92, 49)	140 (92, 48)	169 (112, 57)	137 (91, 48)	136 (91, 45)
Total Group A[2]	109/0	105/0	101/0	83/0	83/0
Total Group B[2]	141/0	140/0	169/0	137/0	136/0
Total No. of Results[3]	1569/0	1566/0	1596/0	1494/0	1882/0
% non -compliant results	**0**	**0**	**0**	**0**	**0**

[1]*Target samples (sampled at harvest, sampled at other stages of production)*
[2] *No. of samples tested/No. of samples non-compliant*
[3]*Total no. of results as samples taken for Group A and Group B substances are tested for multiple residue categories within each group*

4.3.3 Breakdown of 2012-2014 Results

Group A

- **Group A3: Steroids**

Screening for Group A3 was carried out by the Irish Equine Centre (IEC) using an ELISA method. This method screens for methyltestosterone and 17β-oestradiol, in addition it screens for corticosteroids which were previously classed as A3 and now is re-categorised as B2f. Table 14 gives a breakdown of the number of samples tested for in this category in 2012, 2013 and 2014**.**

No non-compliant (i.e. no positive) results were reported for Group A3 samples for the period 2012 to 2014.

Although all samples were compliant (i.e. no positive) for Group A3, in 2012 three samples of farmed salmon from three separate farms and in 2013 two samples of farmed salmon from two separate farms gave a screening reading above the plate cut-off for the Group A3 compound 17β-oestradiol, when these samples were sent for further quantitative LCMSMS screening to the Laboratory of the Government Chemist (LGC) they were found to be compliant.

Table 14: Number of samples tested for Group A3

Year	2012	2013	2014
Methyltestosterone	56	46	44
17β-oestradiol	12	10	10

- **Group A6: Compounds included in Annex IV Council Reg. 2377/90**

Screening for Group A6 was carried out by the Irish Equine Centre (IEC) using an ELISA method and Teagasc Food Research Centre (TFRC) by mass spectrometer. The IEC method screens for chloramphenicol and the TFRC confirmatory method analyses for nitrofurans and nitroimidazole. Table 15 gives a breakdown of the number of samples tested for in this category in 2012, 2013 and 2014**.**

No non-compliant (i.e. no positive) results were reported for Group A6 samples for the period 2012 to 2014.

Table 15: Number of samples tested for Group A6

Year	2012	2013	2014
Chloramphenicol	56	46	44
Nitrofurans*	15	12	12
Nitroimidazoles**	14	12	12

* marker metabolites of the nitrofurans; furazolidone, furaltadone, nitrofurantoin and nitrofurazone

** nitroimidazole metabolites: dimetridazol, ronidazol, metronidazol, hydroxyl-dimetridazol, hydroxyl-metronidazol

Figure 13: SPE clean up technique for analysis

Group B

Group B substances can be classed as authorised substances, unauthorised substances or environmental contaminants.

No non-compliant results were obtained for these compounds for the period 2012-2014.

- **Group B1: Antibacterial Substances**

Antibiotic residues were screened using a qualitative in-house screening method for the detection of quinolones, tetracyclines, sulphonamide and florfenicol using a modification of the Three Plate Test (TPT). In 2013, the Institute developed and validated an Immunoassay qualitative method (Evidence Investigator instrument) for the analysis of sulphonamides which could detect to 50 µg kg^{-1}(i.e. detect to half the MRL), during this period the analysis of sulphonamides was carried out by a subcontract laboratory. In 2014 the testing for sulphonamides was resumed in the Institute and this method obtained INAB accreditation. Table 16 gives a breakdown of the number of samples tested for in this category in 2012, 2013 and 2014.

No non-compliant (i.e. no positive) results were reported for Group B1 samples for the period 2012 to 2014.

Table 16: Number of samples tested for Group B1

Year	2012	2013	2014
Quinolones **Oxolinic acid** **Flumequine**	112	91	91
Tetracyclines **oxytetracycline**	112	91	91
Sulphonamides	112	15*	91**
Florfenicol	112	91	91

*LGC: analysed by UPLC-TOF
**MI: analysed by Evidence Investigator

- **Group B2: Other veterinary drugs**

Group B2 are classed as 'other veterinary drugs'. In aquaculture these veterinary drugs are generally authorised or unauthorised sea lice treatments. Table 17 gives a breakdown of the number of samples tested for in this category in 2012, 2013 and 2014**.**

No non-compliant (i.e. no positive) results were reported for Group B2 samples for the period 2012 to 2014.

Although all samples were compliant in 2014, one sample of freshwater trout gave a screening reading above the plate cut-off for corticosteroids, this sample was found to be compliant when further quantitative LCMS screening by Laboratory of the Government Chemist (LGC) was carried out.

Table 17: Number of samples tested for Group B2

Year	2012	2013	2014
B2a Anthelmintics:			
Ivermectin,	112	91	91
Emamectin B1a	112	91	91
Doramectin	-	-	91
B2c Pyrethroids:			
Cypermethrin	51	91	91
Deltamethrin	86	91	86
B2f – other veterinary drugs:			
Corticosteroids*	37	33	28
B2f other veterinary drugs:			
Teflubenzuron	112	91	91
Diflubenzuron	112	91	91

*dexamethasone, flumethasone and betamethasone

Group B3: Other substances and environmental contaminants

- **Group B3a: Environmental Contaminants Polychlorinated Biphenyls (PCBs):**

As part of the NRCP, the following six non dioxin-like PCBs (NDL-PCB) are monitored; PCB 28, 52, 101, 138, 153, and 180. These NDL-PCBs are routinely used as a monitoring indicator as they are generally presumed to be the most persistent in fish tissue and comprise about half of the amount of total PCB present in feed and food. In the case of NDL-PCBs the

maximum level of 75 µg kg $^{-1}$ wet weight has been set for the sum of these six congeners (also known asICES PCB 6[3]). **None of the samples analysed for the period 2012 to 2014 exceeded this standard of 75 µg kg $^{-1}$ wet weight and were reported as compliant.** Table 18 gives a breakdown of the number of samples tested for in this category in 2012, 2013 and 2014 and Table 20 details the concentration in samples obtained during this period

- **Organochlorine pesticides (OCPs):**

Chlorinated pesticides behave similarly to PCBs in the environment and do not have maximum concentrations in fish set by the EC. A number of OSPAR contracting countries have set levels and Table 11 shows the available standards/guidance values in so far as the Marine Institute is aware of these. **All the samples analysed for chlorinated pesticides for the period 2012 to 2014 were below these levels and were reported as compliant.** Table 18 gives a breakdown of the number of samples tested for in this category in 2012, 2013 and 2014**.**

Table 18: Number of samples tested for Group B3a

Year	2012	2013	2014
PCBs*	22	19	19
OCPs*	12	10	10

*Reference Table 22, 23, 24 for further details

- **Group B3b: Chemical elements**

For the period of 2012 to 2014, levels of mercury, cadmium and lead were all very low and well below the relevant European maximum limits as described in Table 11and **all samples were reported as compliant**. Table 19 gives a breakdown of the number of samples tested for in this category in 2012, 2013 and 2014 and Table 20 details the maximum concentration in samples obtained during this period.

Table 19: Number of samples tested for Group B3b

Year	2012	2013	2014
Mercury	12	10	10
Cadmium	12	10	10
Lead	12	10	10

Table 20: Levels of mercury, cadmium, lead and PCBs during 2012-2014

Year	Maximum concentration	Median (Upper Bound Mean)	Range	EC Maximum Limit	n
mg kg^{-1} ww					
Mercury	0.07 mg kg^{-1}	0.04 (0.04)	<0.02 -0.07	0.5 mg kg^{-1}	32
Cadmium	ND	<0.005 (<0.005)	<0.005	0.05 mg kg^{-1}	32
Lead	0.09 mg kg^{-1}	0.05 (<0.05)	<0.05 – 0.09	0.3 mg kg^{-1}	32
µg kg^{-1} ww					
ICES PCB 6[3]	20.1 µg kg^{-1}	7.59 (7.61)	1.5 -20.1	75 µg kg^{-1}	61

n: number sampled

ICES PCB 6: sum of the following 6 non dioxin like PCBS–PCB 28, 52, 101, 138, 153, 180

- **Group B3c: Mycotoxins**

A mycotoxin is a toxic by-product of mould growth in feed and can remain as a residue in meat tissue. The amount and type of mycotoxin varies with environmental conditions such as temperature and humidity. The NRCP for Aquaculture for the period 2012 to 2014 analysed for the following mycotoxins: aflatoxin B1, aflatoxin B2, aflatoxin G1 and aflatoxin G2.

Aflatoxin B1 is the most common in food and amongst the most potent genotoxic and carcinogenic aflatoxins. The highest aflatoxin concentration obtained for the samples analysed during this period was in 2014 were a concentration of 0.07 μg kg $^{-1}$ (wet weight) for aflatoxin B1 was present, **currently there is no maximum limit set for aflatoxins in fish.**

Table 21: Number of samples tested for Group B3c

Year	2012	2013	2014
Aflatoxins	7	6	6

- **Group B3d: Dyes**

Malachite Green is a common commercial fabric dye which had been widely used both prophylactically and in the treatment of fungal infection of both fish and eggs for over 60 years. It is also effective against several protozoal infestations, including agents causing proliferative kidney disease (PKD) and ichthyophthiriosis (white dot disease). Its use had been primarily associated with freshwater farms and hatcheries; therefore over recent years, monitoring has been scaled up with freshwater installations particularly targeted. Recent results suggest that as a result of increased industry awareness that it is an unauthorised substance, supported by monitoring and enforcement, the use of malachite green has ceased and **all target samples tested for malachite green and its metabolite leuco malachite green for the period 2012 to 2014 were found to be compliant i.e. negative.**

In 2014, in addition to testing for malachite green and its metabolite leuco malachite green a new method and was developed and validated to include brilliant green, crystal violet, leuco crystal violet and victoria blue. These dyes belong to a group of dyes known as triphenylmethane dyes and maybe used illegally in aquaculture as they exhibit antimicrobial and antiparasitic properties. There has been no evidence of brilliant green, crystal violet, leuco crystal violet or victoria blue being used in aquaculture in Ireland; however these dyes have the potential to be used to treat Saprolegnia (fungus) either when present on the fish or as a prophylactic treatment to protect fish eggs from infection. These dyes are believed to have carcinogenic properties and are not authorised for use in the aquaculture industry.

A minimum required performance level (MRPL) has been set for the sum of malachite green and leuco malachite green at 2 μg kg $^{-1}$ and the Marine Institute has set a decision limit of 0.5 μg kg $^{-1}$ for malachite green and leuco malachite green individually i.e. a sample is deemed non-compliant if detected above the decision limit of 0.5 μg kg $^{-1}$. No MRPL has been set for brilliant green, crystal violet, leuco crystal violet and victoria blue, however as these dyes are unauthorised a decision limit of 0.5 μg kg $^{-1}$ has been set for all dyes. **All target samples tested in 2014 for crystal violet and its metabolite leuco crystal violet, brilliant green and victoria blue were found to be compliant i.e. negative.**

Table 22: Number of samples tested for Group B3d

Year	2012	2013	2014
Dyes	86*	79*	69

*Malachite green and leuco malachite green only

Table 23: Summary of 2012 residue monitoring results for target farmed fish samples (salmon and trout). All tests performed on muscle tissue

RESIDUE	GROUP	NUMBER EXAMINED	Non-Compliant[1]	DETECTION LIMIT[2] (µg kg^{-1})
Methyltestosterone	A3	56	0	1.5
17β-oestradiol	A3	12	0	1.5
Chloramphenicol	A6	56	0	0.25
Nitrofurans	A6	15	0	0.06
Nitroimidazole	A6+B2b	14	0	3.0
Tetracyclines: oxytetracycline	B1	112	0	50
Quinolones: Oxolinic acid Flumequine	B1	112	0	75 150
Florfenicol	B1	112	0	250
Emamectin B1a	B2a	112	0	9.0
Ivermectin	B2a	112	0	0.4
Cypermethrin	B2c	51	0	4.0
Deltamethrin	B2c	86	0	4.0
Corticosteroids	B2f	37	0	1.5
Teflubenzuron	B2f	112	0	80
Diflubenzuron	B2f	112	0	86
ICES PCB 6[3]	B3a	22	0	-
Pentachlorobenzene	B3a	12	0	0.02
Hexachlorobenzene	B3a	12	0	0.05
HCP-*cis*	B3a	12	0	0.02
HCP-*trans*	B3a	12	0	0.07
Aldrin	B3a	12	0	0.07
Toxaphene 26	B3a	12	0	0.05
Toxaphene 50	B3a	12	0	0.09
Toxaphene 62	B3a	12	0	0.16
Octachlorstyrene	B3a	12	0	0.01
Dieldrin	B3a	12	0	0.02
Endrin	B3a	12	0	0.03
Mirex	B3a	12	0	0.02
Endosulphane sulphate	B3a	12	0	0.05
Endosulphane- *α*	B3a	12	0	0.07
Endosulphane-*β*	B3a	12	0	0.05
Chlordane-*cis*	B3a	12	0	0.01
Chlordane-*trans*	B3a	12	0	0.01
Nonachlordane-*trans*	B3a	12	0	0.01
α-HCH	B3a	12	0	0.02
β-HCH	B3a	12	0	0.01
γ-HCH	B3a	12	0	0.02
σ-HCH	B3a	12	0	0.08
DDD-o,p'	B3a	12	0	0.02
DDT-o,p'	B3a	12	0	0.02
DDT-p,p'	B3a	12	0	0.02
DDE-o,p'	B3a	12	0	0.01
DDE-p,p'	B3a	12	0	0.01
DDD-p,p'	B3a	12	0	0.02
Lead	B3c	12	0	8
Cadmium	B3c	12	0	2
Mercury	B3c	12	0	8
Aflatoxins	B3d	7	0	0.1
Malachite Green	B3e	86	0	1.0
Leuco Malachite Green	B3e	86	0	1.0

[1] Action limit reference Appendix 2.

[2] Limit of Detection (LOD) for organochlorine compounds are averages as LOD is sample dependant.

[3] ICES PCB 6: sum of the following 6 non dioxin like PCBS–PCB 28, 52, 101, 138, 153, 180. Commission Regulation No 1259/2011 (came into force 1st Jan 2012) amending Regulation No. 1881/2006 *setting maximum levels for dioxins, dioxin-like PCBs and non dioxin-like PCBs in foodstuffs.*

Table 24: Summary of 2013 Residue Monitoring Results for Target farmed fish samples (salmon and trout). All tests performed on muscle tissue

RESIDUE	GROUP	NUMBER EXAMINED	Non-Compliant[1]	DETECTION LIMIT[2] (μg kg $^{-1}$)
Methyltestosterone	A3	46	0	1.5
17β-oestradiol	A3	10	0	1.5
Chloramphenicol	A6	46	0	0.25
Nitrofurans	A6	12	0	0.06
Nitroimidazole	A6+B2b	12	0	3.0
Tetracyclines: oxytetracycline	B1	91	0	50
Quinolones:Oxolinic acid Flumequine	B1	91	0	75 150
Florfenicol	B1	91	0	250
Sulphonamides	B1	15	0	50
Emamectin B1a	B2a	91	0	9.0
Ivermectin	B2a	91	0	0.4
Cypermethrin	B2c	91	0	5
Deltamethrin	B2c	91	0	4
Corticosteroids	B2f	33	0	1.5
Teflubenzuron	B2f	91	0	80
Diflubenzuron	B2f	91	0	86
ICES PCB 6[3]	B3a	19	0	0.61
Pentachlorobenzene	B3a	10	0	0.02
Hexachlorobenzene	B3a	10	0	0.02
cis-Heptachlorepoxide	B3a	10	0	0.02
trans-Heptachlorepoxide	B3a	10	0	0.06
Aldrin	B3a	10	0	0.06
Toxaphene 26	B3a	10	0	0.04
Toxaphene 50	B3a	10	0	0.07
Toxaphene 62	B3a	10	0	0.15
Octachlorstyrene	B3a	10	0	0.02
Dieldrin	B3a	10	0	0.02
Endrin	B3a	10	0	0.02
Mirex	B3a	10	0	0.02
Endosulphan sulphate	B3a	10	0	0.12
α -Endosulphan	B3a	10	0	0.11
β –Endosulphan	B3a	10	0	0.12
cis- Chlordane (α-chlordane)	B3a	10	0	0.02
trans- Chlordane (γ-chlordane)	B3a	10	0	0.02
trans-nonachlor	B3a	10	0	0.02
Oxychlordane	B3a	10	0	0.02
α-HCH	B3a	10	0	0.02
β-HCH	B3a	10	0	0.02
γ-HCH (lindane)	B3a	10	0	0.02
δ -HCH	B3a	10	0	0.02
DDD-o,p'	B3a	10	0	0.02
DDT-o,p'	B3a	10	0	0.02
DDT-p,p'	B3a	10	0	0.02
DDE-o,p'	B3a	10	0	0.02
DDE-p,p'	B3a	10	0	0.02
DDD-p,p'	B3a	10	0	0.02
Lead	B3c	10	0	8
Cadmium	B3c	10	0	2
Mercury	B3c	10	0	8
Aflatoxins	B3d	6	0	0.01
Malachite Green	B3e	79	0	1.0
Leuco Malachite Green	B3e	79	0	1.0

[1] *Action limit reference Appendix 2.*

[2] *Limit of Detection (LOD) for organochlorine compounds are averages as LOD is sample dependant.*

[3] ICES PCB 6: sum of the following 6 non dioxin like PCBS–PCB 28, 52, 101, 138, 153, 180. Commission Regulation No 1259/2011 (came into force 1st Jan 2012) amending Regulation No. 1881/2006 *setting maximum levels for dioxins, dioxin-like PCBs and non dioxin-like PCBs in foodstuffs.*

Table 25: Summary of 2014 Residue Monitoring Results for Target farmed fish samples (salmon and trout). All tests performed on muscle tissue

RESIDUE	GROUP	NUMBER EXAMINED	Non-Compliant[1]	DETECTION LIMIT[2] (µg kg $^{-1}$)
Methyltestosterone	A3	44	0	1.5
17β-oestradiol	A3	10	0	1.5
Chloramphenicol	A6	44	0	0.25
Nitrofurans	A6	12	0	0.06
Nitroimidazole	A6	12	0	3.0
Tetracyclines: oxytetracycline	B1	91	0	50
Quinolones: Oxolinic acid Flumequine	B1	91	0	75 150
Florfenicol	B1	91	0	250
Sulphonamides: Sulphadiazine	B1	91	0	50
Emamectin B1a	B2a	91	0	9.0
Ivermectin	B2a	91	0	0.4
Doramectin	B2a	91	0	0.4
Cypermethrin	B2c	91	0	5
Deltamethrin	B2c	86	0	4
Corticosteroids	B2f	28	0	1.5
Teflubenzuron	B2f	91	0	80
Diflubenzuron	B2f	91	0	86
ICES PCB 6 (incl. LOQ)	B3a	19	0	0.46
DDT (o p')	B3a	10	0	0.02
DDE (o p')	B3a	10	0	0.02
DDD (o p')	B3a	10	0	0.06
DDD (p p')	B3a	10	0	0.06
DDE (p p')	B3a	10	0	0.02
DDT (p p')	B3a	10	0	0.02
hexachlorobenzene	B3a	10	0	0.08
a-HCH	B3a	10	0	0.04
b-HCH	B3a	10	0	0.04
g-HCH (lindane)	B3a	10	0	0.04
d -HCH	B3a	10	0	0.04
Pentachlorobenzene	B3a	10	0	0.08
aldrin	B3a	10	0	0.02
dieldrin	B3a	10	0	0.03
endrin	B3a	10	0	0.05
a-endosulfan	B3a	10	0	0.08
b-endosulfan	B3a	10	0	0.07
endosulfan sulfate	B3a	10	0	0.02
Toxaphene 26	B3a	10	0	0.08
Toxaphene 50	B3a	10	0	0.08
Toxaphene 62	B3a	10	0	0.17
heptachlor	B3a	10	0	0.02
mirex	B3a	10	0	0.02
oxychlordane	B3a	10	0	0.08
cis-heptachlorepoxide	B3a	10	0	0.03
trans-heptachlorepoxide	B3a	10	0	0.05
octachlorostyrene	B3a	10	0	0.01
trans-nonachlor	B3a	10	0	0.01
trans-chlordane (g- chlordane)	B3a	10	0	0.02

Table 25: Summary of 2014 Residue Monitoring Results for Target farmed fish samples (salmon & trout) continued

RESIDUE	GROUP	NUMBER EXAMINED	Non-Compliant[1]	DETECTION LIMIT[2] (μg kg $^{-1}$)
cis-chlordane (a-chlordane)	B3a	10	0	0.02
Lead	B3c	10	0	8
Cadmium	B3c	10	0	2
Mercury	B3c	10	0	8
Aflatoxins	B3d	6	0	0.01
Malachite Green	B3e	69	0	0.5
Leuco Malachite Green	B3e	69	0	0.5
Crystal Violet	B3e	69	0	0.5
Leuco Crystal Violet	B3e	69	0	0.5
Victoria Blue	B3e	69	0	0.5
Brilliant Green	B3e	69	0	0.5

[1] Action limit reference Appendix 1.
[2] Limit of Detection (LOD) for organochlorine compounds are averages as LOD is sample dependant.
[3] ICES PCB 6: sum of the following 6 non dioxin like PCBS–PCB 28, 52, 101, 138, 153, 180. Commission Regulation No 1259/2011 (came into force 1st Jan 2012) amending Regulation No. 1881/2006 *setting maximum levels for dioxins, dioxin-like PCBs and non dioxin-like PCBs in foodstuffs.*

5.0 CONCLUSION

Veterinary treatments, environmental contaminants, unauthorised and banned substances in farmed finfish analysed in 2012 to 2014 were found to be 100% compliant with the requirements of the Residues Directive. **No non-compliant results were detected for samples analyses as part of the routine targeted monitoring**. This suggests that there is continued awareness and improved practices within the industry for the past number of years as there have been no non-compliant results for target samples since 2005 i.e. 100% compliance for surveillance monitoring residue for the period 2006-2014. It also suggests the proper use of medicines and treatments including the observation of extended withdrawal periods.

Concentrations of environmental contaminants such as trace metals were low and well within EC maximum limits. Contaminant levels for PCBs and OCPs analysed as part of 2012 to 2014 NRCP for farmed finfish, were within regulatory available limits where such limits are set for the protection of consumers.

In order to comply with EU legislation, it is necessary that the monitoring of contaminants and veterinary residues in farmed fish continues into the future. In addition to meeting legislative requirements, this monitoring programme provides ongoing assurance of consumer safety and enables ongoing consumer risk assessments. It also provides data to support setting of new and practical standards at international level; and provides factual information as a basis for considered response to food safety concerns relating to farmed finfish. This programme promotes good practice within the fish farming industry on an ongoing basis.

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MAPS

Marine Institute, 2015. Sites Sampled as part of the Residues Programme in 2014. Scale 1:3,157,319, 2015. Using ArcMap 10.2.
Marine Institute, 2015. Sites Sampled as part of the Residues Programme in 2013. Scale 1:3,157,319, 2015. Using ArcMap 10.2.

Marine Institute, 2015. Sites Sampled as part of the Residues Programme in 2012. Scale 1:3,157,319, 2015. Using ArcMap 10.2.

LEGISLATION

This section lists some of the main legal instruments concerning the Irish finfish aquaculture industry for monitoring of NRCP.

European

Council Directive 96/23/EC of 29 April 1996 on measures to monitor certain substances and residues thereof in live animals and animal products and repealing Directives 85/358/EEC and 86/469/EEC and Decisions 89/187/EEC and 91/664/EEC.

Commission Decision 2002/657/EC of 12 August 2002 implementing Council Directive 96/23/EC establishes criteria and procedures for the validation of analytical methods to ensure the quality and comparability of analytical results generated by official laboratories

Commission Decision 2003/181/EC of 13 March 2003 amending Decision 2002/657/EC as regards the setting of minimum required performance limits (MRPLs) for certain residues in food of animal origin.

Commission Decision 2004/25/EC of 22 December 2003 amending Decision 2002/657/EC as regards the setting of minimum required performance limits (MRPLs) for certain residues in food of animal origin

Regulation (EC) No. 882/2004 of the European Parliament and the Council of 29 April 2004 on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules.

Directive 2008/97/EC of the European Parliament and of the Council of 19 November 2008 amending Council Directive 96/22/EC concerning the prohibition on the use in stock farming of certain substances having a hormonal or thyrostatic action and of beta-agonists

REGULATION (EC) No 470/2009 OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 6 May 2009 laying down Community procedures for the establishment of residue limits of pharmacologically active substances in foodstuffs of animal origin, repealing Council Regulation (EEC) No 2377/90 and amending Directive 2001/82/EC of the European Parliament and of the Council and Regulation (EC) No 726/2004 of the European Parliament and of the Council

COMMISSION REGULATION (EU) No 37/2010 of 22 December 2009 on pharmacologically active substances and their classification regarding maximum residue limits in foodstuffs of animal origin

COMMISSION REGULATION (EU) No 420/2011 of 29 April 2011 amending Regulation (EC) No 1881/2006 setting maximum levels for certain contaminants in foodstuffs

COMMISSION REGULATION (EU) No 594/2012 of 5 July 2012 amending Regulation (EC) 1881/2006 as regards the maximum levels of the contaminants ochratoxin A, non dioxin-like PCBs and melamine in foodstuffs

COMMISSION REGULATION (EU) No 1259/2011 of 2 December 2011 amending Regulation (EC) No 1881/2006 as regards maximum levels for dioxins, dioxin-like PCBs and non dioxin-like PCBs in foodstuffs

Commission Implementing Decision 2014/745/EU of 27 October 2011 amending Decision 98/536/EC as regards the list of national reference laboratories

National
Animal Remedies Act, 1993 (Act No 23 of 1993).

European Communities Animal Remedies Regulations 2007. S.I. 786 of 2007 as amended SI12/2009 & SI262/2012

European Communities Control of Animal Remedies and their Residues Regulations 2009. S.I. 183 of 2009 as amended SI263/2012

USEFUL WEBSITES

- BIM www.bim.ie; http://www.bim.ie/about-the-seafood-industry/
- Department of Agriculture, Food and the Marine (Ireland): http://www.agriculture.gov.ie/; http://www.agriculture.gov.ie/animalhealthwelfare/veterinarymedicinesresidues/
- European Agency for the Evaluation of Medicinal Products: http://www.ema.europa.eu/ema; http://www.ema.europa.eu/ema/index.jsp?curl=pages/regulation/landing/veterinary_medicines_regulatory.jsp&mid=WC0b01ac058001ff8a
- European Commission: http://ec.europa.eu/health/veterinary-use/index_en.htm
- European Food Safety Authority: www.efsa.eu
- Food Safety Authority of Ireland: www.fsai.ie; http://www.fsai.ie/legislation/food_legislation/veterinary_medicines/monitoring_of_residues.html
- Marine Institute http://www.marine.ie/Home/; http://www.marine.ie/Home/site-area/areas-activity/seafood-safety/residues-monitoring?language=ga
- Sea Fisheries Protection Authority http://www.sfpa.ie/
- Health Products Regulatory Authority https://www.hpra.ie/

APPENDIX 1: METHODS OF ANALYSIS

Methods of Analysis

1.1 Sample Collection and Preparation

In accordance with the 2014 National Residues Control Plan for Aquaculture under Council Directive 96/23/EC, *Staff authorised under the Animal Remedies Act 1993*, collected samples at farms or at processing plants. All samples were transported to the laboratory under controlled conditions, while ensuring an unbroken chain of custody. Sub-samples were taken for both analytical and archive purposes and all sub-samples were stored frozen (<-20°C).

1.2 Dyes Analysis

Samples were extracted for Dyes analysis with Acetonitrile by shaking in the presence of hydroxylamine and magnesium sulphate. The eluant is evaporated to dryness followed by reconstitution in a mixture of acetonitrile/water /ascorbic acid solution. This solution is centrifuged, filtered and analysed for brilliant green, crystal violet, leuco crystal violet, leuco malachite green, malachite green and victoria blue by Fast Liquid Chromatography coupled to Mass Spectrometry (UFLC-MS/MS).

1.3 Nitrofuran Analysis

The analysis of nitrofurans was carried out by Teagasc Food Research Centre (TFRC). Tissue bound residues of nitrofurans are hydrolysed with acid and derivatised with 2-nitrobenzaldehyde. The nitrophenyl derivatives are extracted with ethyl acetate and determined by Ultra Performance Liquid Chromatography coupled to Mass Spectrometry (UPLC-MS/MS) using deuterated analogues as internal standards for quantification. Metabolites of furazolidone, furaltadone, nitrofurantoin and nitrofurazone are analysed.

1.4 Screening for Group A Compounds & Group B Cortiscosteroids

Group A compounds were screened using the Elisa method, this testing was subcontracted out to Irish Equine Centre (IEC). This method is qualitative in nature and was used to detect residues of 17β-oestradiol, chloramphenicol and methyltesterone. Corticosteroids were also screened using this method.

1.5 Nitroimidazoles

Analysis of nitroimidazoles was carried out by Teagasc Food Research Centre (TFRC). Samples are extracted with acetonitrile, water, magnesium sulphate and sodium chloride; defatted with n-hexane and concentrated. The residue content is determined by Ultra Performance Liquid Chromatography coupled to Mass Spectrometry (UPLC-MS/MS) and analysed for dimetridazole and its metabolite, ipronidazole and its metabolite, metronidazole and its metabolite, ornidazole and ronidazole.

1.6 Analysis for Cypermethrin and Deltramethrin by Gas Chromatography Electron Capture Detection (GC-ECD).

Approximately 2g of sample was extracted using a hexane/acetone mixture, followed by liquid/liquid partition and solid phase extraction techniques. The extract was then evaporated to dryness and reconstituted in 2,2,4-trimethylpentane. The analysis was carried out using Agilent 7890A Gas Chromatography coupled with electron capture detector (GC-ECD) with a Chrompack 15m CpSil 8 column.

1.7 Analysis of Ivermectin, Doramectin and Emamectin B1a by Ultra Fast Liquid Chromatography (UFLC) with Fluorescence Detection.
A representative sample (5g) from each fish was homogenised and extracted with methanol. The extract was cleaned up by liquid/liquid partition and solid phase extraction techniques. The resultant residue was derivatised and analysed by liquid chromatography (UFLC) with fluorescence detection.

1.8 Analysis of Teflubenzuron and Diflubenzuron by Ultra Fast Liquid Chromatography (UFLC) with Ultraviolet (UV) Detection.
This method involves the extraction of approximately 3g of tissue with acetonitrile followed by clean up using liquid/liquid partition and silica SPE. Quantification was carried out by reverse phase UFLC using an acetonitrile/water mobile phase and UV detection. Confirmation and peak purity was evaluated using a photodiode array detector.

1.9 Antimicrobial Screening.
Antimicrobial screening was carried by the Fish Health Unit (FHU) of the Marine Institute, using a modification of the Two Plate Test (TPT). The aim of this method is to reveal residues of substances with antibacterial activity by testing the fish tissue using agar plates that have been seeded with suitably sensitive bacterial cultures. This method is qualitative in nature and was used to detect residues of Quinolones, Tetracyclines, and Florfenicol. Analysis of Sulphonamide was carried out by Immunoassay, this method is qualitative in nature and tested on the Evidence Investigator instrument.

1.10 Analysis for Polychlorinated Biphenyls (PCBs) and Organochlorine Pesticides (OCPs).
Analysis for Polycholorinated Biphenyls (PCBs) and Organochlorine Pesticides (OCPs) was carried out by a subcontracted laboratory (Eurofins). Prior to the extraction, ^{13}C-UL-labeled internal standards were added, followed by an extraction using a solid/lipid extraction and clean up by a multicolumn system. Concentration levels were determined by (high resolution gas chromatography and high resolution mass spectrometry (HRGC/HRMS) using a DB-5 capillary column.

1.11 Determination of Cadmium and Lead using Graphite Furnace Atomic Absorption Spectroscopy (GFAAS).
Concentrated nitric acid (4ml) and hydrogen peroxide (4ml) were added to approximately 0.2g freeze-dried tissue. Samples were then digested with the aid of microwave (CEM Mars5). Lead and cadmium concentrations were determined using Graphite Furnace Atomic Absorption Spectrometry with Zeeman background correction (Varian SpectrAA 220Z).

1.12 Determination of Mercury using Cold Vapour Atomic Fluorescence Spectroscopy (CVAFS).
Concentrated nitric acid (4ml) was added to 0.2g of accurately weighed wet tissue, which was then digested in a laboratory microwave oven (CEM Mars5). Mercury concentrations were determined by Cold Vapour Atomic Fluorescence Spectroscopy (CV-AFS) using a PSA Merlin Analyser.

1.13 Determination of Percentage Moisture Content.

The moisture content was determined by drying approximately 1g of tissue overnight in an oven at 104°C to constant weight.

1.14 Analysis for Mycotoxins.

Analysis of Aflatoxins B1, B2, G1 and G2 was carried out under contract with the LGC UK. The method involved the extraction of about 25g of muscle using dichloromethane and the extract was cleaned up on an immunoaffinity column. The subsequent determination of aflatoxins B1, B2, G1 and G2 was achieved using Liquid Chromatography with Fluorescence Detection after post column derivatisation.

APPENDIX 2: QUALITY CONTROL

To check the quality of the data produced during the 2012-2014 National Surveillance Scheme for chemical residues in farmed fish, Quality Control (QC) samples in the form of either reagent blanks, replicates, spiked samples or Certified Reference Materials (CRMs) were analysed with each batch of samples. The Marine Institute quality assurance results obtained, as shown below ± 1 Standard Deviations (SD), were considered sufficient for the purpose of the monitoring programme. For CRMs, z-scores were calculated using the methodology of QUASIMEME (Quality Assurance of Marine Environment and Monitoring in Europe). A Z-score of between –2 and +2 is generally considered satisfactory for the purpose of environmental monitoring programmes.

QC data for 2012 samples

Analyte	QC Type	Target Value	Result ± SD	Mean Z – Score
• **Anthelmintics (µg kg^{-1})**				
Ivermectin	*Spike (n=18)*	*2.0*	*88.0 ± 5.4% Recovery*	–
Emamectin B1a	*Spike (n=18)*	*100*	*80.0 ± 12.2% Recovery*	–
• **Pyrethroids (µg kg^{-1})**				
Cypermethrin	*Spike (n=20)*	*50*	*90.6 ± 9.4% Recovery*	–
Deltamethrin	*Spike (n=16)*	*10*	*92.3 ± 9.2% Recovery*	–
• **Benzoylurea (µg kg^{-1})**				
Teflubenzuron	*Spike (n=21)*	*500*	*87.8 ± 14.3% Recovery*	–
Diflubenzuron	*Spike (n=21)*	*1000*	*89.0 ± 14.8% Recovery*	–
• **Malachite Green (µg kg^{-1})**				
Malachite Green	*Spike (n=26)*	*2*	*105.8 ± 21.1% Recovery*	–
Leuco Malachite Green	*Spike (n=26)*	*2*	*102.1 ± 19.1% Recovery*	–
• **Chemical Elements (mg kg^{-1})**				
Lead	SRM 2976 (n=2)	1.19	*1.26*	*0.46*
Cadmium	1566b (n=2)	*2.48*	*2.75*	*0.85*
Mercury	DORM 2 (n=1)	*4.64*	*5.70*	*1.79*

QC data for 2013 samples

Analyte	QC Type	Target Value	Result ± SD	Mean Z – Score
• **Benzoylurea (µg kg^{-1})**				
Teflubenzuron	*Spike (n=16)*	*500*	*89.2 ± 9.3% Recovery*	–
Diflubenzuron	*Spike (n=16)*	*1000*	*84.2 ±7.2% Recovery*	–
• **Pyrethroids (µg kg^{-1})**				
Cypermethrin	*Spike (n=20)*	*50*	*81.9 ± 7.9% Recovery*	–
Deltamethrin	*Spike (n=17)*	*10*	*83.9 ± 4.4% Recovery*	–
• **Anthelmintics (µg kg^{-1})**				
New Instrument				
Ivermectin	*Spike (n=11)*	*2*	*89.8 ± 6.2% Recovery*	–
Emamectin B1a	*Spike (n=11)*	*100*	*83.6 ± 10.3% Recovery*	–
Old Instrument				
Ivermectin	*Spike* (n=9)	2	92.8±7.3 % Recovery	--
Emamectin B1a	*Spike* (n=10)	100	88.7±15.3% Recovery	--
• **Malachite Green (µg kg^{-1})**				
Malachite Green	*Spike (n=26)*	*2*	*97.7 ± 9.1% Recovery*	–
Leuco Malachite Green	*Spike (n=27)*	*2*	*101.3 ± 10.3% Recovery*	–
• **Chemical Elements (mg kg^{-1})**				
Lead	CE278K(n=2)	2.18	*2.28*	*0.37*
Cadmium	CE278K (n=1)	*0.336*	*0.32*	*-0.35*
Mercury	CE278K (n=1)	*0.071*	*0.07*	*-0.053*
Mercury	DORM2 (n=1)	*4.64*	*4.826*	*0.315*

QC data for 2014 samples

Analyte	QC Type	Target Value	Result ± SD	Mean Z – Score
• **Benzoylurea (µg kg^{-1})**				
Teflubenzuron	*Spike* (n=25)	500	86.6 ± 7.4% Recovery	--
Diflubenzuron	*Spike* (n=27)	1000	80.3 ± 6.2% Recovery	--
• **Pyrethroids (µg kg^{-1})**				
Cypermethrin	*Spike* (n=18)	50	82.0 ± 8.0% Recovery	--
Deltamethrin	*Spike* (n=16)	10	84.9 ± 7.7% Recovery	--
• **Anthelmintics (µg kg^{-1})**				
Ivermectin	*Spike* (n=22)	2	87.5 ± 6.7% Recovery	--
Emamectin B1a	*Spike* (n=22)	100	79.8 ± 7.6% Recovery	--
Doramectin	*Spike* (n=22)	2	92.7 ± 8.1%Recovery	--
• **Dyes (µg kg^{-1})**				
Brilliant Green	*Spike* (n=31)	2	97.8 ± 13.2% Recovery	--
Crystal Violet	*Spike* (n=31)	2	99.6 ± 3.4% Recovery	--
Leuco Crystal Violet	*Spike* (n=31)	2	100.9 ± 6.8%Recovery	--
Leuco Malachite Green	*Spike* (n=29)	2	107.6±13.3% Recovery	--
Malachite Green	*Spike* (n=31)	2	101.4 ±7.0% Recovery	--
Victoria Blue	*Spike* (n=31)	2	102.8±11.2% Recovery	--
• **Dry weight (%)**	QTMO58BT (n=1)	23.56	102.0 % Recovery	0.16
• **Chemical Elements (mg kg^{-1} dry weight)**				
Lead	SRM 2976 (n=1)	1.19	124.9% Recovery	1.96
Cadmium	SRM 2976 (n=1)	0.82	117.6% Recovery	1.28
Mercury	DORM2 (n=2)	4.64	113.4% Recovery	1.05

APPENDIX 3: 2012, 2013, 2014 PLANS FOR THE MONITORING AND DETECTION OF RESIDUES IN AQUACULTURE PRODUCTS

2012 Plan for the Monitoring and Detection of Residues in Aquaculture products

1. National Legislation on use of substances listed in Annex I of Directive 96/23/EC

Animal Remedies Act, 1993 (No. 23 of 1993)
Animal Remedies Regulations, 2007 (SI No. 786 of 2007)
Control of Animal Remedies and their Residues Regulations 2009(SI No. 183 of 2009)

2. Relevant Departments and their infrastructure

Dept of Agriculture, Food and the Marine
Agriculture House
Kildare Street
Dublin 2

Sea Fisheries Protection Authority
Block B
Clogheen
Clonakilty
Co. Cork

Marine Institute
Rinville
Oranmore
Co. Galway

3. Staff resources to carry out plan

Authorised Officers will collect all samples.

Group A substances will be performed by the Irish Equine Centre- Kildare, Laboratory of the Government Chemist-UK, Ashtown Food Research Centre-Dublin and the CRL -Bilthoven

Analyses for Group B substances will be performed within the Marine Institute with the exception of what is indicated in the plan.

4. Approved laboratories

Marine Institute,
Rinville,
Oranmore,
Co. Galway.

Laboratory of the Government Chemist,
Queens Road,
Teddington,
Middlesex,
TW11 OLY,
England

Irish Equine Centre (IEC),
Johnstown,
Naas,
Co. Kildare.

RIKILT-CRL
Laboratory for Residue analysis,
NL-3720 BA BILTHOVEN,
1) Netherland,

Eurofins GfA GmbH,
D-48161 Münster,
Germany

Ashtown Food Research Centre,
Teagasc,
Ashtown
Dublin 15

Wessling GmbH,
Bochum,
Germany

5. Additional Information

For Group A analysis more than half the samples are 'on farm' samples, taken at various stages of production, the remainder are samples taken at harvest.

DIRECTIVE 96/23/EC ANNUAL PLAN FOR THE EXAMINATION FOR RESIDUES IN FARMED FINFISH FOR THE YEAR 2012

Sampling levels and frequency:
Minimum number of finfish from which samples must be taken in 2012.

Total Tonnes Produced 2010	Total min. no. to be tested**	Min. no. Group A	Min. no. Group B
16,793	Production (tonnes)/100=168	1/3 Total Tested= 56	2/3 Total Tested= 112

** min no. to be tested will be based on 2010 finfish production numbers as production numbers not available for 2011

Details of Group A substances monitored in 2012

1 Group of Substances	2 Compounds	3 Matrix	4 Laboratory Method	5 CCbeta (screening) Detection capability	6 CCalpha (confirmatory) decision limit	7 Level of action	8 Number of samples	9 Laboratory
GROUP A								
A 3 Steroids[ж]	Methyltestosterone	Muscle & Skin	(1) ELISA (2) GCMS	1)1.5 µg kg^{-1}	2)0.5 µg kg^{-1}	Presence	56*	(1) IEC (2) CRL
	17 ß oestradiol	Muscle & Skin	(1) ELISA∞ (2) GCMS	1)1.5 µg kg^{-1} 1)1.0 µg kg^{-1}∞	2)0.5 µg kg^{-1}	0.5 µg kg^{-1}	12*	(1) IEC∞ (2) CRL
A 6 Compounds included in Annex IV Council Reg. 2377/90	Chloramphenicol	Muscle & Skin	(1) ELISA∞ (2) GCMS	1)0.25 µg kg^{-1} 1)0.3 µg kg^{-1}∞	2)0.19 µg kg^{-1}	Presence	56*	(1) IEC∞ (2) LGC
	Nitrofurans AOZ AMOZ AHD SEM	Muscle & Skin	LCMSMS	-	0.5 µg kg^{-1} 0.5 µg kg^{-1} 1.0 µg kg^{-1} 1.0 µg kg^{-1}	Presence	14*	AFC
	Nitroimidazoles Dimetridazol hydroxyl- Dimetridazol Ronidazol Metronidazol hydroxyl- Metronidazol	Muscle & Skin	LCMSMS	-	2.2 µg kg^{-1} 1.1 µg kg^{-1} 3.0 µg kg^{-1} 2.6 µg kg^{-1} 2.9 µg kg^{-1}	Presence	14*	LGC

* At least 50% of Group A are "on farm" samples

Column 4: Screening is No. (1) and Confirmatory is No. (2)

∞For screened positive samples for Chloramphenicol using the Elisa, these samples will be sent to subcontract laboratory LGC for further screening (LCMS).

ж Corticosteroids: re-categorised as B2f

Details of Group B substances monitored in 2012

1 Group of Substances	2 Compounds	3 Tissue	4 Laboratory Method	5 CCbeta (screening) Detection capability	6 CCalpha (confirmatory) decision limit	7 Level of action	8 Number of samples	9 Laboratory
B 1 Antibacterial substances	Microbiological screening: Quinolones: -Oxolinic acid -Flumequine Tetracyclines: -oxytetracycline Sulphonamides -Sulphadiazine Florfenicol	Muscle & Skin	Modified EC 3-plate method.	Zone size ≥ 2mm	N/A	>MRL	112	Marine Institute
	Tetracycline Chlortetracycline Epi-Chlortetracycline Oxytetracycline Epi-Oxytetracycline Tetracycline Epi-Tetracycline Doxycycline	Muscle & Skin	1)LC-TOF 2)LCMSMS	 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1}	 120 µg kg^{-1} 135 µg kg^{-1} 124 µg kg^{-1} 130 µg kg^{-1} 122 µg kg^{-1} 146 µg kg^{-1} 135 µg kg^{-1}	 120 µg kg^{-1} 135 µg kg^{-1} 124 µg kg^{-1} 130 µg kg^{-1} 122 µg kg^{-1} 146 µg kg^{-1} 135 µg kg^{-1}	Confirmation and post screening identification of positive Microbiological Samples	LGC

Details of Group B substances monitored in 2012 cont.

1	2	3	4	5	6	7	8	9
Group of Substances	**Compounds**	**Tissue**	**Laboratory Method**	**CCbeta (screening) Detection capability**	**CCalpha (confirmatory) decision limit**	**Level of action**	**Number of samples**	**Laboratory**
	Quinolones							LGC
	Ciprofloxacin		1)LC-TOF 2)LCMSMS	50 µg kg^{-1}	128 µg kg^{-1}	128 µg kg^{-1}		
	Enrofloxacin			50 µg kg^{-1}	125 µg kg^{-1}	125 µg kg^{-1}		
	Danofloxacin			50 µg kg^{-1}	134 µg kg^{-1}	134 µg kg^{-1}		
	Difloxacin			150 µg kg^{-1}	401 µg kg^{-1}	401 µg kg^{-1}		
	Flumequine			200 µg kg^{-1}	700 µg kg^{-1}	700 µg kg^{-1}		
	Oxolinic acid			50 µg kg^{-1}	124 µg kg^{-1}	124 µg kg^{-1}		
	Sarafloxacin			50 µg kg^{-1}	40 µg kg^{-1}	40 µg kg^{-1}		

Column 4: Screening is No. (1) and Confirmatory is No. (2)

Details of Group B substances monitored in 2012 cont.

1	2	3	4	5	6	7	8	9
Group of Substances	**Compounds**	**Tissue**	**Laboratory Method**	**CCbeta (screening) Detection capability**	**CCalpha (confirmatory) decision limit**	**Level of action**	**Number of samples**	**Laboratory**
	<u>Sulphonamides</u> Sulphathiazole Sulphaquinoxaline Sulphapyridine Sulphamethoxy-pyridazine Sulphamonomethoxine Sulphamethazine Sulphamerazine Sulphisoxazole Sulphadimethoxine Sulphadiazine Sulphachlorpyridazine Sulphamethizole		1)LC-TOF 2)LCMSMS	 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1}	**	**		LGC
B2 Other veterinary drugs								
B2 (a) Anthelmintics	Ivermectin Emamectin B1a	Muscle & Skin	HPLC-Flu	- -	0.4 µg kg^{-1} 124 µg kg^{-1}	0.4 µg kg^{-1} 124 µg kg^{-1}	112	Marine Institute
B2(b) Nitroimidazoles^{++}	hydroxyl-ipronidazole		LCMSMS	-	2.2 µg kg^{-1}	Presence	14*	LGC
B2 (c) Carbamates / Pyrethroids	Cypermethrin Deltamethrin		1)GC-ECD 2)GC-MS	1)43 µg kg^{-1} 1)8 µg kg^{-1}	2)65 µg kg^{-1} 2)15 µg kg^{-1}	65 µg kg^{-1} 15 µg kg^{-1}	112	1)Marine Institute 2)LGC

Details of Group B substances monitored in 2012 cont.

1	2	3	4	5	6	7	8	9
Group of Substances	**Compounds**	**Tissue**	**Laboratory Method**	**CCbeta (screening) Detection capability**	**CCalpha (confirmatory) decision limit**	**Level of action**	**Number of samples**	**Laboratory**
B2 (f) Other Pharmacologically active substances	Teflubenzuron Diflubenzuron		HPLC-DAD	- -	583 µg kg^{-1} 1172 µg kg^{-1}	583 µg kg^{-1} 1172 µg kg^{-1}	112	Marine Institute
	Corticosteroids Betamethasone Dexamethasone Flumethasone		(1)ELISA (2) LC-MS	1.5 µg kg^{-1} 1.5 µg kg^{-1} 1.5 µg kg^{-1}	0.5 µg kg^{-1} 0.5 µg kg^{-1} 0.5 µg kg^{-1}	0.5 µg kg^{-1} 0.5 µg kg^{-1} 0.5 µg kg^{-1}	33*	(1) IEC* (2) CRL

* At least 50% are "on farm" samples

Column 4: Screening is No. (1) and Confirmatory is No. (2)

**LGC has an accredited procedure for the development and validation of methods in place in the event that a fish sample tested screen positive using the LC-TOF method. CC alpha will be calculated at that point

++Included in testing for nitromidazoles see A6 compounds

* For screened positive samples for Corticosteroids using the Elisa, these samples will be sent to subcontract laboratory LGC for further screening (LCMS).

Details of Group B substances monitored in 2012 cont.

1	2	3	4	5	6	7	8
Group of Substances	**Compounds**	**Tissue**	**Laboratory Method**	**Detection limit**	**Level of action**	**Number of samples**	**Laboratory**
B3 Other substances and environmental contaminants							
B3 (a) Organochlorine compounds including PCBs	**PCBs** CB Congener 28 CB Congener 52 CB Congener 101 CB Congener 118 CB Congener 138 CB Congener 153 CB Congener 180	Muscle & Skin	GCHRMS	 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1}	Excess of Guideline value	23	Eurofins
	Chlorinated Pest. α-HCH β-HCH γ-HCH δ-HCH DDT-o,p' DDT-p,p' DDD-o,p' DDD-p,p' DDE-o,p' DDE-p,p' HCB Aldrin Dieldrin Endrin *cis*-Chlordane		GCHRMS	 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1}	Excess of Guideline value	12	

Details of Group B substances monitored in 2012 cont.

1	2	3	4	5	6	7	8
Group of Substances	**Compounds**	**Tissue**	**Laboratory Method**	**Detection limit**	**Level of action**	**Number of samples**	**Laboratory**
	trans-Chlordane *oxy*chlordane *trans*-Nonachlordane			0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1}			
B3 (c) Chemical elements	Lead Cadmium Mercury		GFAAS GFAAS CVAFS	8 µg kg^{-1} 2 µg kg^{-1} 8 µg kg^{-1}	300 µg kg^{-1} 50 µg kg^{-1} 500 µg kg^{-1}	12 12 12	Marine Institute
B3 (d) Mycotoxins	Aflatoxin B1 Aflatoxin B2 Aflatoxin G1 Aflatoxin G2	Muscle & Skin	HPLC-Flu	0.01 µg kg^{-1} 0.01 µg kg^{-1} 0.01 µg kg^{-1} 0.01 µg kg^{-1}	-	7	Wessling

Details of Group B3(e) substances monitored in 2012

1	2	3	4	5	6	7	8	9
Group of Substances	**Compounds**	**Tissue**	**Laboratory Method**	**CCbeta (screening) Detection capability**	**CCalpha (confirmatory) decision limit**	**Level of action**	**Number of samples**	**Laboratory**
B3 (e) Dyes	Malachite Green (MG) Leuco Malachite Green (LMG)		LCMSMS	- -	1.0 µg kg^{-1}## 1.0 µg kg^{-1}##	1.0 µg kg^{-1}##	85# (21 salmon/sea trout, 16 Freshwater trout, 48 salmon smolts)	Marine Institute

#64 of the 85 samples for malachite green and leuco malachite green are "on farm" samples.

Reporting limit for MG and LMG

2013 Plan for the Monitoring and Detection of Residues in Aquaculture products

1. **National Legislation on use of substances listed in Annex I of Directive 96/23/EC**
   Animal Remedies Act, 1993 (No. 23 of 1993)
   Animal Remedies Regulations, 2007 (SI No. 786 of 2007)
   Control of Animal Remedies and their Residues Regulations 2009(SI No. 183 of 2009)

2. **Relevant Departments and their infrastructure**
   Dept of Agriculture, Food and the Marine
   Agriculture House
   Kildare Street
   Dublin 2

   Sea Fisheries Protection Authority
   Block B
   Clogheen
   Clonakilty
   Co. Cork

   Marine Institute
   Rinville
   Oranmore
   Co. Galway

3. **Staff resources to carry out plan**
   Authorised Officers will collect all samples.

   Group A substances will be performed by the Irish Equine Centre- Kildare, Laboratory of the Government Chemist-UK, Ashtown Food Research Centre-Dublin and the CRL -Bilthoven

   Analyses for Group B substances will be performed within the Marine Institute with the exception of what is indicated in the plan.

4. **Approved laboratories**
   Marine Institute,
   Rinville,
   Oranmore,
   Co. Galway.

   Laboratory of the Government Chemist,
   Queens Road,
   Teddington,
   Middlesex,
   TW11 OLY,
   England

   Irish Equine Centre (IEC),
   Johnstown,
   Naas,
   Co. Kildare.

   RIKILT-CRL
   Laboratory for Residue analysis,
   NL-3720 BA BILTHOVEN,
   Netherland,

   Eurofins GfA GmbH,
   D-48161 Münster,
   Germany

Ashtown Food Research Centre,
Teagasc,
Ashtown
Dublin 15

Wessling GmbH,
Bochum,
Germany

5. Additional Information

For Group A analysis more than half the samples are 'on farm' samples, taken at various stages of production, the remainder are samples taken at harvest.

DIRECTIVE 96/23/EC ANNUAL PLAN FOR THE EXAMINATION FOR RESIDUES IN FARMED FINFISH FOR THE YEAR 2013

Sampling levels and frequency:
Minimum number of finfish from which samples must be taken in 2013.

Total Tonnes Produced 2011	Total min. no. to be tested**	Min. no. Group A	Min. no. Group B
13,396	Production (tonnes)/100=134	1/3 Total Tested= 45	2/3 Total Tested= 89

** min. no. to be tested will be based on 2011 finfish production numbers as production numbers not available for 2012

Details of Group A substances monitored in 2013

1 Group of Substances	2 Compounds	3 Matrix	4 Laboratory Method	5 CCbeta (screening) Detection capability	6 CCalpha (confirmatory) decision limit	7 Level of action	8 Number of samples	9 Laboratory
GROUP A								
A 3 Steroids[ж]	Methyltestosterone	Muscle & Skin	(1) ELISA (2) GCMS	1)1.5 µg kg^{-1}	2)0.5 µg kg^{-1}	Presence	46*	(1) IEC (2) CRL
	17 Betaoestradiol	Muscle & Skin	(1) ELISA[∞] (2) GCMS	1)1.5 µg kg^{-1} 1)1.0 µg kg^{-1}[∞]	2)0.5 µg kg^{-1}	0.5 µg kg^{-1}	10*	(1) IEC[∞] (2) CRL
A 6 Compounds included in Annex IV Council Reg. 2377/90	Chloramphenicol	Muscle & Skin	(1) ELISA[∞] (2) GCMS	1)0.25 µg kg^{-1} 1)0.3 µg kg^{-1}[∞]	2)0.19 µg kg^{-1}	Presence	46*	(1) IEC[∞] (2) LGC
	Nitrofurans AOZ AMOZ AHD SEM	Muscle & Skin	LCMSMS	-	0.5 µg kg^{-1} 0.5 µg kg^{-1} 1.0 µg kg^{-1} 1.0 µg kg^{-1}	Presence	12*	AFC
	Nitroimidazoles Dimetridazol hydroxyl- Dimetridazol Ronidazol Metronidazol hydroxyl- Metronidazol	Muscle & Skin	LCMSMS	-	2.2 µg kg^{-1} 1.1 µg kg^{-1} 3.0 µg kg^{-1} 2.6 µg kg^{-1} 2.9 µg kg^{-1}	Presence	12*	LGC

* At least 50% of Group A are "on farm" samples

Column 4: Screening is No. (1) and Confirmatory is No. (2)

[∞]For screened positive samples for Chloramphenicol and 17 Betaoestradiol using the Elisa, these samples will be sent to subcontract laboratory LGC for further screening (LCMS).

[ж] Corticosteroids: re-categorised as B2f

Details of Group B substances monitored in 2013

1 Group of Substances	2 Compounds	3 Tissue	4 Laboratory Method	5 CCbeta (screening) Detection capability	6 CCalpha (confirmatory) decision limit	7 Level of action	8 Number of samples	9 Laboratory
B 1 Antibacterial substances	Microbiological screening: Quinolones: -Oxolinic acid -Flumequine Tetracyclines: -oxytetracycline Florfenicol	Muscle & Skin	Modified EC 3-plate method.	75 150 50 250	N/A	>MRL	89	Marine Institute
	Screening: Sulphonamides -Sulphadiazine		1)LC-TOF	50 $\mu g\ kg^{-1}$	N/A	>MRL	15	LGC
	Tetracycline Chlortetracycline Epi-Chlortetracycline Oxytetracycline Epi-Oxytetracycline Tetracycline Epi-Tetracycline Doxycycline	Muscle & Skin	1)LC-TOF 2)LCMSMS	 50 $\mu g\ kg^{-1}$ 50 $\mu g\ kg^{-1}$ 50 $\mu g\ kg^{-1}$ 50 $\mu g\ kg^{-1}$ 50 $\mu g\ kg^{-1}$ 50 $\mu g\ kg^{-1}$ 50 $\mu g\ kg^{-1}$	 120 $\mu g\ kg^{-1}$ 135 $\mu g\ kg^{-1}$ 124 $\mu g\ kg^{-1}$ 130 $\mu g\ kg^{-1}$ 122 $\mu g\ kg^{-1}$ 146 $\mu g\ kg^{-1}$ 135 $\mu g\ kg^{-1}$	 120 $\mu g\ kg^{-1}$ 135 $\mu g\ kg^{-1}$ 124 $\mu g\ kg^{-1}$ 130 $\mu g\ kg^{-1}$ 122 $\mu g\ kg^{-1}$ 146 $\mu g\ kg^{-1}$ 135 $\mu g\ kg^{-1}$	Confirmation and post screening identification of positive Microbiological Samples	LGC

Details of Group B substances monitored in 2013 cont.

1	2	3	4	5	6	7	8	9
Group of Substances	**Compounds**	**Tissue**	**Laboratory Method**	**CCbeta (screening) Detection capability**	**CCalpha (confirmatory) decision limit**	**Level of action**	**Number of samples**	**Laboratory**
	Quinolones							LGC
	Ciprofloxacin Enrofloxacin Danofloxacin Difloxacin Flumequine Oxolinic acid Sarafloxacin		1)LC-TOF 2)LCMSMS	50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 150 µg kg^{-1} 200 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1}	128 µg kg^{-1} 125 µg kg^{-1} 134 µg kg^{-1} 401 µg kg^{-1} 700 µg kg^{-1} 124 µg kg^{-1} 40 µg kg^{-1}	128 µg kg^{-1} 125 µg kg^{-1} 134 µg kg^{-1} 401 µg kg^{-1} 700 µg kg^{-1} 124 µg kg^{-1} 40 µg kg^{-1}		

Column 4: Screening is No. (1) and Confirmatory is No. (2)

Details of Group B substances monitored in 2013 cont.

1	2	3	4	5	6	7	8	9
Group of Substances	**Compounds**	**Tissue**	**Laboratory Method**	**CCbeta (screening) Detection capability**	**CCalpha (confirmatory) decision limit**	**Level of action**	**Number of samples**	**Laboratory**
	<u>Sulphonamides</u> Sulphathiazole Sulphaquinoxaline Sulphapyridine Sulphamethoxy-pyridazine Sulphamonomethoxine Sulphamethazine Sulphamerazine Sulphisoxazole Sulphadimethoxine Sulphadiazine Sulphachlorpyridazine Sulphamethizole		2)LCMSMS	 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1}	**	**		LGC
B2 Other veterinary drugs								
B2 (a) Anthelmintics	Ivermectin Emamectin B1a	Muscle & Skin	HPLC-Flu	- -	0.4 µg kg^{-1} 124 µg kg^{-1}	0.4 µg kg^{-1} 124 µg kg^{-1}	89	Marine Institute
B2(b) Nitroimidazoles++	hydroxyl-ipronidazole		LCMSMS	-	2.2 µg kg^{-1}	Presence	12*	LGC
B2 (c) Carbamates / Pyrethroids	Cypermethrin Deltamethrin		1)GC-ECD 2)GC-MS	1)43 µg kg^{-1} 1)8 µg kg^{-1}	2)65 µg kg^{-1} 2)15 µg kg^{-1}	65 µg kg^{-1} 15 µg kg^{-1}	89	1)Marine Institute 2)LGC

Details of Group B substances monitored in 2013 cont.

1	2	3	4	5	6	7	8	9
Group of Substances	**Compounds**	**Tissue**	**Laboratory Method**	**CCbeta (screening) Detection capability**	**CCalpha (confirmatory) decision limit**	**Level of action**	**Number of samples**	**Laboratory**
B2 (f) Other Pharmacologically active substances	Teflubenzuron		HPLC-DAD	-	583 µg kg^{-1}	583 µg kg^{-1}	89	Marine
	Diflubenzuron			-	1172 µg kg^{-1}	1172 µg kg^{-1}		Institute
	Corticosteroids Betamethasone Dexamethasone Flumethasone		(1)ELISA (2) LC-MS	1.5 µg kg^{-1} 1.5 µg kg^{-1} 1.5 µg kg^{-1}	0.5 µg kg^{-1} 0.5 µg kg^{-1} 0.5 µg kg^{-1}	0.5 µg kg^{-1} 0.5 µg kg^{-1} 0.5 µg kg^{-1}	30*	(1) IEC^ (2) CRL

* At least 50% are "on farm" samples

Column 4: Screening is No. (1) and Confirmatory is No. (2)

**LGC has an accredited procedure for the development and validation of methods in place in the event that a fish sample tested screen positive using the LC-TOF method. CC alpha will be calculated at that point

++Included in testing for nitromidazoles see A6 compounds

^ For screened positive samples for Corticosteroids using the Elisa, these samples will be sent to subcontract laboratory LGC for further screening (LCMS).

Details of Group B3 substances monitored in 2013

1	2	3	4	5	6	7	8
Group of Substances	Compounds	Tissue	Laboratory Method	Detection limit	Level of action	Number of samples	Laboratory
B3 Other substances and environmental contaminants							
B3 (a) Organochlorine compounds including PCBs	PCBs CB Congener 28 CB Congener 52 CB Congener 101 CB Congener 118 CB Congener 138 CB Congener 153 CB Congener 180 Chlorinated Pest. α-HCH β-HCH γ-HCH δ-HCH DDT-o,p' DDT-p,p' DDD-o,p' DDD-p,p' DDE-o,p' DDE-p,p' HCB	Muscle & Skin	GCHRMS	 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1}	Excess of Guideline value	19	Eurofins
	Aldrin Dieldrin Endrin *cis*-Chlordane *trans*-Chlordane *oxy*chlordane *trans*-Nonachlordane		GCHRMS	0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1}	Excess of Guideline value	10	

Details of Group B3 substances monitored in 2013 cont.

1	2	3	4	5	6	7	8
Group of Substances	Compounds	Tissue	Laboratory Method	Detection limit	Level of action	Number of samples	Laboratory
B3 (c) Chemical elements	Lead		GFAAS	8 µg kg $^{-1}$	300 µg kg $^{-1}$	10	Marine Institute
	Cadmium		GFAAS	2 µg kg $^{-1}$	50 µg kg $^{-1}$	10	
	Mercury		CVAFS	8 µg kg $^{-1}$	500 µg kg $^{-1}$	10	
B3 (d) Mycotoxins	Aflatoxin B1	Muscle & Skin	HPLC-Flu	0.01 µg kg^{-1}	-	6	Wessling
	Aflatoxin B2			0.01 µg kg^{-1}			
	Aflatoxin G1			0.01 µg kg^{-1}			
	Aflatoxin G2			0.01 µg kg^{-1}			

Details of Group B3(e) substances monitored in 2013

1	2	3	4	5	6	7	8	9
Group of Substances	**Compounds**	**Tissue**	**Laboratory Method**	**CCbeta (screening) Detection capability**	**CCalpha (confirmatory) decision limit**	**Level of action**	**Number of samples**	**Laboratory**
B3 (e) Dyes	Malachite Green (MG) Leuco Malachite Green (LMG)		LCMSMS	- -	1.0 µg kg^{-1}## 1.0 µg kg^{-1}##	1.0 µg kg^{-1}##	79# (17 salmon/sea trout, 16 Freshwater trout, 46 salmon smolts)	Marine Institute

#62 of the 79 samples for malachite green and leuco malachite green are "on farm" samples.
Reporting limit for MG and LMG

2014 Plan for the Monitoring and Detection of Residues in Aquaculture products

1. **National Legislation on use of substances listed in Annex I of Directive 96/23/EC**
   Animal Remedies Act, 1993 (No. 23 of 1993)
   Animal Remedies Regulations, 2007 (SI No. 786 of 2007)
   Control of Animal Remedies and their Residues Regulations 2009(SI No. 183 of 2009)

2. **Relevant Departments and their infrastructure**
   Dept of Agriculture, Food and the Marine
   Agriculture House
   Kildare Street
   Dublin 2

   Sea Fisheries Protection Authority
   Block B
   Clogheen
   Clonakilty
   Co. Cork

   Marine Institute
   Rinville
   Oranmore
   Co. Galway

3. **Staff resources to carry out plan**
   Authorised Officers will collect all samples.

   Group A substances will be performed by the Irish Equine Centre- Kildare, Laboratory of the Government Chemist-UK, Ashtown Food Research Centre-Dublin and the CRL -Bilthoven

   Analyses for Group B substances will be performed within the Marine Institute with the exception of those indicated in the plan.

4. **Approved laboratories**
   Marine Institute,
   Rinville,
   Oranmore,
   Co. Galway.

   Laboratory of the Government Chemist,
   Queens Road,
   Teddington,
   Middlesex,
   TW11 OLY,
   England

   Irish Equine Centre (IEC),
   Johnstown,
   Naas,
   Co. Kildare.

   RIKILT-CRL
   Laboratory for Residue analysis,
   NL-3720 BA BILTHOVEN,
   Netherland,

   Eurofins GfA GmbH,
   D-48161 Münster,
   Germany

Ashtown Food Research Centre,
Teagasc,
Ashtown
Dublin 15

Wessling GmbH,
Bochum,
Germany

5. Additional Information

For Group A analysis more than half the samples are 'on farm' samples, taken at various stages of production, the remainder are samples taken at harvest.

DIRECTIVE 96/23/EC ANNUAL PLAN FOR THE EXAMINATION FOR RESIDUES IN FARMED FINFISH FOR THE YEAR 2014

Sampling levels and frequency:

Minimum number of finfish from which samples must be taken in 2014.

Total Tonnes Produced 2012	Total min. no. to be tested**	Min. no. Group A	Min. no. Group B
13,221	Production (tonnes)/100=132	1/3 Total Tested= 44	2/3 Total Tested= 88

** min. no. to be tested will be based on 2012 finfish production numbers as production numbers not available for 2013

Details of Group A substances monitored in 2014

1 Group of Substances	2 Compounds	3 Matrix	4 Laboratory Method	5 CCbeta (screening) Detection capability	6 CCalpha (confirmatory) decision limit	7 Level of action	8 Number of samples	9 Laboratory
GROUP A								
A 3 Steroids[ж]	Methyltestosterone	Muscle & Skin	(1) ELISA (2) GCMS	1)1.5 µg kg^{-1}	2)0.5 µg kg^{-1}	Presence	44*	(1) IEC (2) CRL
	17 Betaoestradiol	Muscle & Skin	(1) ELISA[∞] (2) GCMS	1)1.5 µg kg^{-1} 1)1.0 µg kg^{-1}[∞]	2)0.5 µg kg^{-1}	0.5 µg kg^{-1}	9*	(1) IEC[∞] (2) CRL
A 6 Compounds included in Annex IV Council Reg. 2377/90	**Chloramphenicol**	Muscle & Skin	(1) ELISA[∞] (2) GCMS	1)0.25 µg kg^{-1} 1)0.3 µg kg^{-1}[∞]	2)0.19 µg kg^{-1}	Presence	44*	(1) IEC[∞] (2) LGC
	Nitrofurans AOZ AMOZ AHD *SEM*	Muscle & Skin	LCMSMS	-	0.04 1µg kg^{-1} 0.061 µg kg^{-1} 0.057 µg kg^{-1} 0.064 µg kg^{-1}	Presence	11*	AFC
	Nitroimidazoles Dimetridazole HMMNI Metronidazole Hydroxyl- Metronidazole Ornidazole Ronidazole Ipronidazole Hydroxyl-ipronidazole	Muscle & Skin	LCMSMS	-	0.12 µg kg^{-1} 1.0 µg kg^{-1} 0.08 µg kg^{-1} 0.15 µg kg^{-1} 0.29 µg kg^{-1} 0.10 µg kg^{-1} 0.15 µg kg^{-1} 0.09 µg kg^{-1}	Presence	11*	AFC

* At least 50% of Group A are "on farm" samples

Column 4: Screening is No. (1) and Confirmatory is No. (2)

[∞]For screened positive samples for Chloramphenicol and 17 Betaoestradiol using the Elisa, these samples will be sent to subcontract laboratory LGC for further screening (LCMS).

[ж] Corticosteroids: re-categorised as B2f

Details of Group B substances monitored in 2014

1 Group of Substances	2 Compounds	3 Tissue	4 Laboratory Method	5 CCbeta (screening) Detection capability	6 CCalpha (confirmatory) decision limit	7 Level of action	8 Number of samples	9 Laboratory
B 1 Antibacterial substances	Microbiological screening: Quinolones: -Oxolinic acid -Flumequine Tetracyclines: -oxytetracycline Florfenicol	Muscle & Skin	Modified EC 3-plate method.	 75 150 50 250	N/A	>MRL	91	Marine Institute
	Screening: Sulphonamides -Sulphadiazine	Muscle & Skin	1)Immuno assay $	50 µg kg^{-1}	N/A	>MRL	81	Marine Institute
	Tetracycline Chlortetracycline Epi-Chlortetracycline Oxytetracycline Epi-Oxytetracycline Tetracycline Epi-Tetracycline Doxycycline	Muscle & Skin	1)LC-TOF 2)LCMSMS	 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1}	 120 µg kg^{-1} 135 µg kg^{-1} 124 µg kg^{-1} 130 µg kg^{-1} 122 µg kg^{-1} 146 µg kg^{-1} 135 µg kg^{-1}	 120 µg kg^{-1} 135 µg kg^{-1} 124 µg kg^{-1} 130 µg kg^{-1} 122 µg kg^{-1} 146 µg kg^{-1} 135 µg kg^{-1}	Confirmation and post screening identification of positive Microbiological Samples	LGC

Details of Group B substances monitored in 2014 cont.

1 Group of Substances	2 Compounds	3 Tissue	4 Laboratory Method	5 CCbeta (screening) Detection capability	6 CCalpha (confirmatory) decision limit	7 Level of action	8 Number of samples	9 Laboratory
	Quinolones							LGC
	Ciprofloxacin		1)LC-TOF 2)LCMSMS	50 µg kg^{-1}	128 µg kg^{-1}	128 µg kg^{-1}		
	Enrofloxacin			50 µg kg^{-1}	125 µg kg^{-1}	125 µg kg^{-1}		
	Danofloxacin			50 µg kg^{-1}	134 µg kg^{-1}	134 µg kg^{-1}		
	Difloxacin			150 µg kg^{-1}	401 µg kg^{-1}	401 µg kg^{-1}		
	Flumequine			200 µg kg^{-1}	700 µg kg^{-1}	700 µg kg^{-1}		
	Oxolinic acid			50 µg kg^{-1}	124 µg kg^{-1}	124 µg kg^{-1}		
	Sarafloxacin			50 µg kg^{-1}	40 µg kg^{-1}	40 µg kg^{-1}		

Column 4: Screening is No. (1) and Confirmatory is No. (2)
[$] Method validated 2013, INAB audit due in May 2014

Details of Group B substances monitored in 2014 cont.

1 Group of Substances	2 Compounds	3 Tissue	4 Laboratory Method	5 CCbeta (screening) Detection capability	6 CCalpha (confirmatory) decision limit	7 Level of action	8 Number of samples	9 Laboratory
	Sulphonamides Sulphathiazole Sulphaquinoxaline Sulphapyridine Sulphamethoxy-pyridazine Sulphamonomethoxine Sulphamethazine Sulphamerazine Sulphisoxazole Sulphadimethoxine Sulphadiazine Sulphachlorpyridazine Sulphamethizole	Muscle & Skin	2)LCMSMS	 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1} 50 µg kg^{-1}	**	**		LGC
B2 Other veterinary drugs								
B2 (a) Anthelmintics	Ivermectin Emamectin B1a Doramectin[$]	Muscle & Skin	UFLC-Flu	- - -	0.4 µg kg^{-1} 129 µg kg^{-1} 0.4 µg kg^{-1}	0.4 µg kg^{-1} 129 µg kg^{-1} 0.4 µg kg^{-1}	91	Marine Institute
B2 (c) Carbamates / Pyrethroids	Cypermethrin Deltamethrin		1)GC-ECD 2)GC-MS	1)41 µg kg^{-1} 1)9 µg kg^{-1}	2)65 µg kg^{-1} 2)15 µg kg^{-1}	65 µg kg^{-1} 15 µg kg^{-1}	91	1)Marine Institute 2)LGC
B2 (f) Other Pharmacologically active substances	Teflubenzuron		UFLC-DAD	-	572 µg kg^{-1}	572 µg kg^{-1}	91	Marine Institute

Details of Group B substances monitored in 2014 cont.

1	2	3	4	5	6	7	8	9
Group of Substances	**Compounds**	**Tissue**	**Laboratory Method**	**CCbeta (screening) Detection capability**	**CCalpha (confirmatory) decision limit**	**Level of action**	**Number of samples**	**Laboratory**
	Diflubenzuron			-	1119 µg kg^{-1}	1119 µg kg^{-1}		
	Corticosteroids Betamethasone Dexamethasone Flumethasone		(1)ELISA (2) LC-MS	1.5 µg kg^{-1} 1.5 µg kg^{-1} 1.5 µg kg^{-1}	0.5 µg kg^{-1} 0.5 µg kg^{-1} 0.5 µg kg^{-1}	0.5 µg kg^{-1} 0.5 µg kg^{-1} 0.5 µg kg^{-1}	28*	(1) IEC^K (2) CRL

* At least 50% are "on farm" samples

Column 4: Screening is No. (1) and Confirmatory is No. (2)

**LGC has an accredited procedure for the development and validation of methods in place in the event that a fish sample tested screen positive using the LC-TOF method. CC alpha will be calculated at that point

^K For screened positive samples for Corticosteroids using the Elisa, these samples will be sent to subcontract laboratory LGC for further screening (LCMS).

$ Method validated 2013, INAB audit due in May 2014

Details of Group B3 substances monitored in 2014

1	2	3	4	5	6	7	8
Group of Substances	**Compounds**	**Tissue**	**Laboratory Method**	**Detection limit**	**Level of action**	**Number of samples**	**Laboratory**
B3 Other substances and environmental contaminants							
B3 (a) Organochlorine compounds including PCBs	**PCBs** CB Congener 28 CB Congener 52 CB Congener 101 CB Congener 118 CB Congener 138 CB Congener 153 CB Congener 180	Muscle & Skin	GCHRMS	 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1}	Excess of Guideline value	19	Eurofins
	Chlorinated Pest. α-HCH β-HCH γ-HCH δ-HCH DDT-o,p' DDT-p,p' DDD-o,p' DDD-p,p' DDE-o,p' DDE-p,p' HCB Aldrin Dieldrin Endrin *cis*-Chlordane *trans*-Chlordane *oxy*chlordane *trans*-Nonachlordane		GCHRMS	 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1} 0.1 µg kg^{-1}	Excess of Guideline value	10	

Details of Group B3 substances monitored in 2014 cont.

1	2	3	4	5	6	7	8
Group of Substances	**Compounds**	**Tissue**	**Laboratory Method**	**Detection limit**	**Level of action**	**Number of samples**	**Laboratory**
B3 (c) Chemical elements	Lead		GFAAS	8 µg kg^{-1}	300 µg kg^{-1}	10	Marine Institute
	Cadmium		GFAAS	2 µg kg^{-1}	50 µg kg^{-1}	10	
	Mercury		CVAFS	8 µg kg^{-1}	500 µg kg^{-1}	10	
B3 (d) Mycotoxins	Aflatoxin B1	Muscle & Skin	HPLC-Flu	0.01 µg kg^{-1}	-	6	Wessling
	Aflatoxin B2			0.01 µg kg^{-1}			
	Aflatoxin G1			0.01 µg kg^{-1}			
	Aflatoxin G2			0.01 µg kg^{-1}			

Details of Group B3(e) substances monitored in 2014

1 Group of Substances	2 Compounds	3 Tissue	4 Laboratory Method	5 CCbeta (screening) Detection capability	6 CCalpha (confirmatory) decision limit	7 Level of action	8 Number of samples	9 Laboratory
B3 (e) Dyes	Malachite Green (MG) Leuco Malachite Green (LMG) Brillant Green (BG) Crystal Violet (CV) Leuco Crystal Violet (LCV) Victoria Blue (VB)	Muscle & Skin	LCMSMS[$]	- - 	0.5 µg kg^{-1} 0.5 µg kg^{-1} 0.5 µg kg^{-1} 0.5 µg kg^{-1} 0.5 µg kg^{-1} 0.5 µg kg^{-1}	0.5 µg kg^{-1} 0.5 µg kg^{-1} 0.5 µg kg^{-1} 0.5 µg kg^{-1} 0.5 µg kg^{-1} 0.5 µg kg	69# (17 salmon/sea trout, 8 Freshwater trout harvest, 8 Freshwater trout osop, 36 salmon smolts)	Marine Institute

#52 of the 69 samples for dyes are "on farm" samples.

[$] Method validated 2013, INAB audit due in May 2014

APPENDIX 4: LIST OF SAMPLING LOCATIONS FROM 2012 TO 2014

Company	Site	Species	Stage of Production	Date
Delphi Fishery Ltd.	Delphi Lodge	Salmon	Other Stage of Production	2014
Derrylea Holdings Ltd.	Lough Fee	Salmon	Other Stage of Production	2014
ESB Fisheries Conservation	Ballyshannon	Salmon	Other Stage of Production	2014
Marine Harvest Ireland	Lough Altan	Salmon	Other Stage of Production	2014
Marine Harvest Ireland	Roancarraig	Salmon	Harvest	2014
Salmo Nova Ltd.	Garryhill	Salmon	Other Stage of Production	2014
Derrylea Holdings Ltd.	Poulmounty Salmon Hatchery	Salmon	Other Stage of Production	2014
Bradan Beo Teo	Dinish	Salmon	Harvest	2014
Marine Harvest Ireland	Roancarraig	Salmon	Harvest	2014
Bradan Beo Teo	Dinish	Salmon	Harvest	2014
Marine Harvest Ireland	Roancarraig	Salmon	Harvest	2014
Mannin Bay Salmon Co. Ltd	Curhownagh	Salmon	Harvest	2014
Curraun Fisheries Ltd.	Mulranny	Seawater Trout	Harvest	2014
Mannin Bay Salmon Co. Ltd	Curhownagh	Salmon	Harvest	2014
Ocean Farm Ltd.	McSwynes Bay	Salmon	Harvest	2014
Bradan Beo Teo	Dinish	Salmon	Harvest	2014
Cullion Fish Farm	Cullion	Freshwater Trout	Other Stage of Production	2014
Raford Trout Farm	Kiltullagh	Freshwater Trout	Harvest	2014
Araglen Valley Trout Farm	Araglen	Freshwater Trout	Other Stage of Production	2014
Murphys Irish Seafood	Gerahies	Salmon	Harvest	2014
Inland Fisheries Ireland	Roscrea	Freshwater Trout	Harvest	2014
Ocean Farm Ltd.	McSwynes Bay	Salmon	Harvest	2014
Douglas Valley Hatchery	Kilclough	Salmon	Other Stage of Production	2014
Marine Harvest Ireland	Rinmore (Deenish)	Salmon	Harvest	2014

List of Sampling Locations From 2012 to 2014

Company	Site	Species	Stage of Production	Date
Marine Harvest Ireland	Creevin (Inver)	Salmon	Harvest	2014
Derrylea Holdings Ltd.	Poulmounty	Salmon	Other Stage of Production	2014
Marine Harvest Ireland	Ardmore	Salmon	Harvest	2014
Mannin Bay Salmon Co. Ltd	Leitir Aird	Salmon	Harvest	2014
Marine Harvest Ireland	Deenish	Salmon	Harvest	2014
Marine Harvest Ireland	Lough Altan	Salmon	OSOP	2013
Marine Harvest Ireland	Millbrook	Salmon	OSOP	2013
Marine Harvest Ireland	Pettigo	Salmon	OSOP	2013
Derrylea Holdings	Screebe	Salmon	OSOP	2013
Western Regional Fisheries Board	Cong	Salmon	OSOP	2013
Marine Institute	Furnace	Salmon	OSOP	2013
Goatsbridge	Goatsbridge	Freshwater Trout	OSOP	2013
IDAS Woodenbridge	Aughrim	Freshwater Trout	OSOP	2013
Murphys Irish Seafood	Borlin Hatchery	Salmon	OSOP	2013
Santa Cruise	Santa Cruise	Salmon	OSOP	2013
Western Regional Fisheries Board	Cong	Salmon	OSOP	2013
Mannin Bay Salmon Co. Ltd	Curhownagh	Salmon	Harvest	2013
Marine Harvest Ireland	Inishfanard	Salmon	Harvest	2013
Eany Fish Products Ltd.	Inver Bay	Seawater Trout	Harvest	2013
Marine Harvest Ireland	Ahabeg	Salmon	Harvest	2013
Mannin Bay Salmon Co. Ltd	Curhownagh	Salmon	Harvest	2013
Marine Harvest Ireland	Ahabeg	Salmon	Harvest	2013
Ocean Farm	Inver Bay	Salmon	Harvest	2013
Murphys Irish Seafood ltd.	Gearhies	Salmon	Harvest	2013
Goatsbridge	Goatsbridge	Freshwater Trout	Harvest	2013

List of Sampling Locations From 2012 to 2014

Company	Site	Species	Stage of Production	Date
Inland Fisheries Ireland	Roscrea Fish Farm	Freshwater Trout	Harvest	2013
Marine Harvest Ireland	Inishfanard	Salmon	Harvest	2013
Mannin Bay Salmon Co. Ltd	Curhownagh	Salmon	Harvest	2013
Marine Harvest Ireland	Glinsk	Salmon	Harvest	2013
Mannin Bay Salmon Co. Ltd	Rosroe	Salmon	Harvest	2013
Ocean Farm Ltd.	Ocean Farm Processing Plant	Salmon	Harvest	2013
Marine Harvest Ireland	Glinsk	Salmon	Harvest	2013
Marine Harvest Ireland	Inishfanard	Salmon	Harvest	2013
Mannin Bay Salmon Co. Ltd	Rosroe	Salmon	Harvest	2013
Marine Harvest Ireland	Inishfanard	Salmon	Harvest	2013
Marine Harvest Ireland	Killary	Salmon	Harvest	2012
Marine Harvest Ireland	Inver	Salmon	Harvest	2012
Curran Blue Ltd.	Clew Bay	Seawater Trout	Harvest	2012
Derrylea Holdings Ltd.	Lough Ahalie	Salmon	OSOP	2012
Marine Harvest Ireland	Lough Altan	Salmon	OSOP	2012
Marine Harvest Ireland	Millbrook	Salmon	OSOP	2012
Marine Harvest Ireland	Pettigo	Salmon	OSOP	2012
Mannin Bay Salmon Co. Ltd	Ballinakill	Salmon	Harvest	2012
Delphi Fishery Ltd.	Leenane	Salmon	OSOP	2012
Parteen Salmon Hatchery	Parteen	Salmon	OSOP	2012
Salmo Nova Ltd.	Garryhill	Salmon	OSOP	2012
Eany Fish Products Ltd.	Inver	Salmon	Harvest	2012
Marine Harvest Ireland	Killary	Salmon	Harvest	2012
Marine Harvest Ireland	Inishfanard	Salmon	Harvest	2012
Eany Fish Products Ltd.	Inver	Seawater Trout	Harvest	2012

List of Sampling Locations From 2012 to 2014

Company	Site	Species	Stage of Production	Date
Ocean Farm Ltd.	Mc Swynes Bay (Site: 84C)	Salmon	Harvest	2012
Meitheal Eisc Teo	Dinish	Salmon	Harvest	2012
Marine Harvest Ireland	Roancarraig	Salmon	Harvest	2012
Mannin Bay Salmon Co. Ltd	Curhownagh	Salmon	Harvest	2012
Araglen Valley Trout Farm	Araglen Valley Trout Farm	Freshwater Trout	Harvest	2012
Araglen Valley Trout Farm	Araglen Valley Trout Farm	Freshwater Trout	OSOP	2012
Murphys Irish Seafood Ltd.	Gearhies	Salmon	Harvest	2012
IDAS Trout Ltd.	Aughrim Farm	Freshwater Trout	OSOP	2012
IDAS Trout Ltd.	Woddenbridge	Freshwater Trout	Harvest	2012
Marine Harvest Ireland	Roancarraig	Salmon	Harvest	2012
Curraun Fisheries Ltd.	Mulranny	Seawater Trout	Harvest	2012
Douglas Valley Hatchery	Douglas Valley	Salmon	OSOP	2012
Murphys Irish Seafood Ltd.	Derrynafinchin Borlin	Salmon	OSOP	2012
Marine Harvest Ireland	Fanad - Swilly	Salmon	Harvest	2012
Marine Harvest Ireland	Tievetooey, Pettigo	Salmon	OSOP	2012
Mannin Bay Salmon Co. Ltd	Curhownagh	Salmon	Harvest	2012
Ocean Farm Ltd.	Mc Swynes Bay	Salmon	Harvest	2012
Marine Harvest Ireland	Swilly	Salmon	Harvest	2012
Marine Harvest Ireland	Swilly	Salmon	Harvest	2012
Marine Harvest Ireland	Inishfanard	Salmon	Harvest	2012

LIST OF ACRONYMS

AFC - Ashtown Food Centre
CRM's - Certified Reference Materials
CVAFS - Cold Vapour Atomic Fluorescence Spectroscopy
DAFM - Department of Agriculture, Fisheries & the Marine
DDD - Dichlorodiphenyldichloroethane
DDE - Dichlorodiphenyldichloroethylene
DDT - Dichlorodiphenyltrichloroethane
EC – European Commission
EEC – European Economic Community
EFSA – European Food Safety Authority
ELISA method - Enzyme-Linked ImmunoSorbent Assay
ESB - Electricity Supply Board
EU – European Union
FHU - Fish Health Unit
FSAI - Food Safety Authority of Ireland
GC-ECD - Gas Chromatography Electron Capture Detection
GFAAS - Graphite Furnace Atomic Absorption Spectroscopy
HBCD - Hexabromocyclododecane
HCB - Hexachlorobenzene
HCH - Hexachlorocyclohexane
HPLC - Performance Liquid Chromatography
HPRA – Health Products Regulatory Authority
HRGC - High Resolution Gas Chromatography
HRMS - High Resolution Mass Spectrometry
ICES - International Council for Exploration of the Sea
IEC - Irish Equine Centre
INAB - Irish National Accreditation Board
ISO - International Organization for Standardization
ISPG - Irish Seafood Producers Group
LCMSMS - Liquid Chromatography with tandem Mass Spectrometry detection
LGC, UK - Laboratory of the Government Chemist, United Kingdom
LMG - Leuco Malachite Green
LOD - Limit of Detection
MG - Malachite Green
MH - Marine Harvest Ireland Ltd.
MRL - Maximum Residue Limit
NDL-PCB non dioxin-like Polychlorinated Biphenyls
NRCP - National Residue Control Plan
NRL - National Reference Laboratory
OCP's - Organochlorine pesticides
OSOP - Other Stages of Production
OSPAR - OSPAR Commission, protecting and conserving the North-East Atlantic and its resources
PCB - Polychlorinated Biphenyls
PDBE - Polybrominated diphenyl ethers
POP - Persistant organic pollutants
QC - Quality Control
QUASIMEME - Quality Assurance of Marine Environment and Monitoring in Europe
SFPA – Sea Fisheries Protection Authority
SI - Statutory Instrument
SPE - Solid Phase Extraction
TPT - Three Plate Test
UV - Ultraviolet
VIs - Veterinary Inspector's

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